# PRACTICAL EMULSIONS

A Handbook of Emulsions, Emulsifiers
and Methods of Formulating and
Making Emulsions of Practical
Value in Industry

By

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1943

CHEMICAL PUBLISHING CO., INC. BROOKLYN, N. Y. U. S. A.

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#### PREFACE.

ALTHOUGH there are many excellent books on colloid chemistry, which deal with emulsions in a theoretical and general manner, there is none which specifically deals with emulsions for the practical worker. This book, therefore, attempts to touch only lightly on the theoretical aspects and concentrates on the art of making and applying emulsions. No attempt has been made to compile a complete record of all published work on emulsions. The attempt has been rather to give sufficiently diverse examples to illustrate the advances in this field.

Because of certain desirable properties, emulsions are used in many varied fields. A few examples are wax and oil emulsions used as polishes; soluble oils in metal cutting; fatliquors in leather processing; oil emulsions in textile treatment; agricultural sprays, water-paints, food, medicinal products, and so forth.

The greatest drawback to the increased use of emulsions is the lack of a proper understanding of the technique and formulation that is necessary to produce a good emulsion. While most formulations in this field have been obtained by "rule-of-thumb" methods, they are predicated on innumerable experiments and have withstood the test of long usage.

The purpose of this book is to set forth this practical information, within small compass, thus making it readily available to technical workers, who it is hoped, will find it helpful.

H. BENNETT.

#### INTRODUCTION

Although emulsions are finding newer and wider applications each day, basically they are not new. In 1674 a paper was read before the Royal Society, describing experiments whereby oils, resins and gums were incorporated in vinous and aqueous media.<sup>1</sup>

An emulsion, for practical purposes, is a dispersion of small drops of one liquid in another liquid, each of which is insoluble in the other. Pure mutually insoluble liquids will not usually emulsify. Consequently, certain natural impurities or the addition of a third material (called the emulsifying agent) is necessary to effect emulsification.

A colloidal solution is a dispersion of one phase in another as definite grains, globules, bubbles, filaments or films where the latter have at least one dimension of 5–200 millimicrons. While many emulsions are colloidal, others containing large globules are not.

Dispersed particles, less than 3 microns in diameter, exhibit a continuous, rapid, oscillating motion when observed through a suitable microscope, a phenomenon known as Brownian movement.

Hardy, Langmuir and Harkins developed the idea of the disposition of an emulsifying agent at the interface of two

<sup>&</sup>lt;sup>1</sup> Chemist and Druggist, 132, 475 (1940).

immiscible liquids. In the dineric interface the molecules are arranged regularly rather than haphazardly so that similar, groups face each other. Thus, the organic radical of the emulsifier orients itself toward the organic phase (oil, solvent or wax) whereas the active or polar groups turn toward the aqueous phase. In an organic material having polar groups there is a great affinity for water with a resultant lower surface tension and a tendency toward increased miscibility. The following are examples of polar groups: CN, COOH, COOM, COOR, NH<sub>2</sub>, NHCH<sub>3</sub>, NCS, COR, CHO, OH, NO<sub>2</sub>, C1, Br, I and groups having N, S, O, and double bonds. (M=metal; R=radical).

This knowledge induced the synthesis of thousands of compounds, of varying degrees of complexity, which act as emulsifying agents. New emulsifying agents are being created each day as research develops new chemical compounds.

The hydrodynamics of emulsification, *i.e.*, the mechanical forces used to produce emulsions, is exceedingly complex and is not thoroughly understood. Since these forces are applied most un-uniformly it can be readily seen that such variables will give variable results. It is exceedingly important, in attempting to duplicate known emulsions, that all forces be duplicated as closely as possible.

The mechanics of emulsification concerns itself with two opposing tendencies.<sup>2</sup> The globules are broken down in size by impact and shearing forces. These tend to form unstable laminae or cylinders which in turn tend to coalesce. These tendencies are affected, to varying degrees, by interfacial tension, volume ratio, viscosity and density of the phases.

When mechanical forces are weak, interfacial tension plays an important part. When stresses are high, as in an homo-

<sup>&</sup>lt;sup>2</sup> Stamm, J., Phys. Chem., 30, 996 (1930).

genizer, interfacial tension plays only a minor role. In either case, it may have considerable influence in globule coalescence where the disperse phase has a great volume. During the emulsification and after the emulsion has been formed, coalescence takes place.

A usual prerequisite for emulsification is adsorption in one phase and solution in the other, the interface being composed of the emulsifier.<sup>3</sup> The interface generally consists of more than one layer of molecules.<sup>4</sup>

There are many theories on emulsions and each has its adherents. Until the disputants can agree, there is not much point in discussing the merits of each theory.

<sup>&</sup>lt;sup>3</sup> Freundlich, et al., Technical Aspects of Emulsions, Chemical Publishing Co., Inc. (1936). <sup>4</sup> McBain, J., Amer. Chem. Soc., 49, 2230 (1927); 51, 3534 (1939).

# CONTENTS

C <b>H</b> APTER		PAGE
	Preface	. iii
	Introduction	. v
	Part I General	
I.	Emulsifying Agents	. 1
II.	Types of Emulsions and Foams	. 14
III.	METHODS, FORMULATION, EQUIPMENT	. 37
IV.	STABILITY	. 56
V.	GENERAL TECHNICAL EMULSIONS	. 80
VI.	DISPERSING AGENTS, WETTING AGENTS.	. 103
VII.	Emulsifying Agents, List of	. 114
VIII.	Emulsions, List of	. 145
IX.	DEMULSIFYING AND DEFOAMING AGENTS,	
	List of	. 197
	Part II Formulas	
I.	Agricultural Spray Emulsions	. 203
II.	CUTTING OILS, SOLUBLE OILS, MISCIBLE	
	Oils	. 216
III.	Emulsifying Agents	. 227
IV.	BITUMINOUS EMULSIONS	. 232
	ix	

Contents

 $\mathbf{x}$ 

CHAPTER				PAGE
V.	CLEANERS AND SOAPS			239
VI.	COSMETIC AND DRUG EMULSIONS			251
VII.	Defoaming Agents			294
VIII.	FOOD EMULSIONS			297
IX.	Gasoline Emulsions			308
X.	LACQUER EMULSIONS			313
XI.	LEATHER TREATMENT EMULSIONS			320
XII.	LUBRICANT EMULSIONS			325
XIII.	MEDICINAL EMULSIONS			328
XIV.	PAINT EMULSIONS			340
XV.	PAPER PROCESSING EMULSIONS .			348
XVI.	Polish Emulsions			354
XVII.	RESIN AND RUBBER EMULSIONS			413
XVIII.	Textile Emulsions			423
XIX.	WATERPROOFING EMULSIONS .			433
XX.	Wax Emulsions			436
XXI.	MISCELLANEOUS			448
XXII.	Dispersions			450
VVIII	INDEX			453

# PART I GENERAL

Linseed extract for margarine 10 and bituminous emulsions. 11 Humates for bituminous emulsions,12 tannic acid 13 or rubber latex 14 for bituminous emulsions. Lecithin for margarine 15 and bitumen emulsion, 18 Pectin 17 for emulsions of insecticidal and edible oils as well as egg yolk.18

An emulsifying agent need not necessarily lower surface tension (e.g., Saponin) but it must form a protective film around each globule.19

The controlling factor, determining the type of dynamic dispersion, is the relative volume of oil and water. The internal pressure of liquids probably controls the effect of volume ratio. The dispersion type can usually be predicted from the relation between internal pressure and surface tension of pure liquids. Systems containing less than 25% water usually form water-in-oil dispersions, while systems containing more than 31% water usually form oil-in-water dispersions. In extreme cases, systems containing less than 10% water form water-in-oil dispersions, systems containing more than 45% water form oil-in-water dipersions, and systems containing 10-45% water may form either type. Between 10 and 45% water content, the factors that exert the chief effect in determining the type of dispersion are the relative specific gravities, viscosities and dielectric constants of the oil and water, the pH of the water, and the effects of emulsifying

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10 Blichfeldt, Brit. 104,899 (1916).
11 Schutte, U. S. 1,950,272 (1934).
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<sup>&</sup>lt;sup>12</sup> I. G., Brit. 369,242 (1930); I. G., Brit. 334,426 (1928); I. G., Brit. 317,496 (1928); Ges. F. T., Ger. 591,340 (1934).

<sup>18</sup> Levy, Brit. 246,907 (1924).

<sup>&</sup>lt;sup>14</sup> Coles, Brit. 329,965 (1929); Coles, U. S. 1,886,334 (1932).

<sup>&</sup>lt;sup>15</sup> Behrend, Brit. 405,116 (1933); Bollman, Brit. 369,990 (1930).

<sup>&</sup>lt;sup>16</sup> Muhlenwerke, Brit. 383,432 (1931).

<sup>&</sup>lt;sup>17</sup> Deiglmayr, Ger. 585,586; 575,922 (1934); Calif., Fr. Gr. Ex. Brit. 369,518 (1930); Patterson, U. S. 1,726,364 (1929); Douglas, Brit. 221,466.

18 Bergsvik, U. S. 1,941,243 (1933); Hellerud, U. S. 1,941,261 (1933).

Brit. 341,414 (1929); Heide, Brit. 357,246 (1930).

<sup>&</sup>lt;sup>19</sup> Hillyer, Z., physik. Chem. 47, 336 (1904).

agents. In a relatively narrow transition zone, the character and intensity of agitation determine the type of dispersion. Less important and often negligible factors are the shape and size of the container, total volume of the liquids, preferential wetting ability of the container walls, surface-tension, differential and the interfacial tension. The relations between dispersion and emulsification were investigated with crude oil and brine, which form stable water-in-oil emulsions. The dispersion characteristics of the system coincide with the general characteristics of dispersions, they are independent of the method of emulsification dependence but apparently control it to a considerable extent. Intermittent agitation is 20 times more efficient than continuous agitation because it suppresses the dispersion of oil in water. The limiting concentration of stable emulsions is approximately 50% with excess brine and continuous agitation, and 75% with intermittent agitation and addition of the brine in increments.1

The power of any emulsifying agent and dispersion factor can be calculated from comparison of quantities of undispersed phase and unused dispersion medium in identical emulsions.<sup>2</sup>

Emulsifiers have been classified as products of C, H, and O; C, H, O, and S; C, H, O, and N; and are rated by their effect in emulsions.<sup>2a</sup>

An electrical charge on the oil phase particles, formed by adsorption of OH (from water) ions, may substitute the emulsifying agent.<sup>8</sup>

The particle size of ion stabilized emulsions is about 0.4 microns regardless of method of preparation.4

The emulsifying power of sodium stearate and sodium

<sup>&</sup>lt;sup>1</sup> Rottseig, J., Phys. Chem. (U.S.S.R.) 9, 35 (1937).

Smith, J., Soc. Chem. Ind. 46, 345 (1937).
 Gallo, Boll. chim. farm. 77, 517 (1938).

<sup>&</sup>lt;sup>3</sup> Thomas, Jol. Ind. & Eng. Chem. 12, 177 (1920).

<sup>4</sup> Lewis, Kolloid-Z-4, 211 (1909).

palmitate <sup>5</sup> has been compared by determination of the smallest quantity of soap necessary to emulsify equal volumes of different oils. The purity of the soaps was very important. The final volume of soap solution and oil used for each emulsion was 100 cc. The curves (weight of soap in grams with cc. of oil) of cottonseed and poppy-seed oils were similar, as were those of mustard-seed, sesame, olive and coconut oils. With each increase of 10 cc. of oil the amount of soap increased, although this increase was not in direct proportion, i.e., the emulsifying power of the soap increased with increase in concentration. In general, the emulsifying power of sodium palmitate was greater than that of sodium stearate. The soap solution with the lower surface tension possessed the greater emulsifying power.

The sodium, potassium and ethanolamine soaps prepared from purified naphthenic acid <sup>6</sup> are appreciably less alkaline in reaction than other soaps; their aqueous solutions, even in high concentrations, do not tend to gel; and they are excellent emulsifying agents.

Oil-in-water emulsions 7 are given by sodium oleate, arabic acid; arabates of sodium, magnesium and iron; valerates of ammonium and sodium; sodium glyconate, galactonate, dihydroxystearate, salicylate and gallate. Water-in-oil emulsions are given by ursolic acid, 64 oleates of magnesium, calcium, cobalt (ous); magnesium and aluminum; magnesium and zinc valerates or dihydroxystearate. Lead arabate does not serve, and the following substances give both types: calcium gluconate, cadmium galactonate, calcium salicylate and gallate. In emulsions of vegetable or mineral oil with acacia,

<sup>&</sup>lt;sup>5</sup> Jajnix Kolloid-Z-37, 139 (1925); White, J., Phys. Chem. 24, 617 (1920).

<sup>&</sup>lt;sup>6</sup> Minich, Textile Colorist, 60, 698 (1938).

 <sup>&</sup>lt;sup>7</sup> Krantz, Colloid Symposium Monograph 6, 173 (1928).
 <sup>6a</sup> Carleton, The Chemist, 18, 453 (1941).

the greatest stability exists when the pH of the external phase is between 2 and 10. With tragacanth, the pH range is 1.9 to 2.3. Greatest stability for acacia is at a pH of 4.11 to 4.28; for tragacanth, about 2.5. Particle size, surface tension, interfacial tension and viscosity were determined at various pH. Particle size and viscosity in tragacanth emulsions are affected by pH but not in acacia emulsions. However particle size increased in the latter with pH over 7.

Olive oil gives a more stable emulsion with magnesium oleate than does mineral oil, the most stable emulsion range being pH 11–12.5, with an extremely unstable range at pH 2.5. Surface tension of water-in-oil emulsions is unaffected by pH changes of the internal phase; unstable emulsions have a lower viscosity than stable ones; particle-size variations, up to 30 microns average, do not alter stability.

The active emulsifiers of lanolin and lanolin alcohols 8 have been represented as "hydroxycholesterol" and "metacholesterol". Both of these are mixtures of sterols and are only partially precipitated by digitonin. The active principle of these mixtures is cholesterol and its emulsifying power is somewhat increased by the presence of fatty alcohols in the unsaponified portion of wool fat. Pure isocholesterol has no emulsifying properties. The removal of isocholesterol from lanolin alcohols, increases their emulsifying power. The emulsifying action of hexadecanol is superior to hexacosanol. B Cholestanol is slightly inferior to cholesterol. Pure cholestandiol has poor emulsifying power even when mixed with cholesterol. α Hydroxycholesterol, β hydroxycholesterol and cholestanolon have good emulsion properties. Cholestantriol has no emulsifying properties. Of the zoosterols, coprosterol is as good as dihydrocholesterol, of which it is an isomer. In

<sup>&</sup>lt;sup>8</sup> Janistyn, Fette U. Seifen 47, 351 (1940); Lifsohvetz, Ber. 29, 2890; Roemer, J. Amer. Phar. Assoc. 1, 673.

#### Practical Emulsions

general, sterols precipitated by digitonin have emulsifying properties.

Experiments were conducted on the emulsification power of some fatty alcohol-solvents combinations 9 to determine (1) the number of cc. of oil which can be held in emulsion by 1 cc. of the aqueous solutions and (2) the "half-value" time at which half the emulsion had been separated. In these experiments, which were conducted at room temperature, 0.2% solutions and consistent amounts of solvent were taken. The emulsification period was 5 minutes. Fresh textile preparations were used and 0.2 cc. of oil was added at a time. Results: Using a soap solution, soap solvent, Turkey-red oil solution, fatty alcohol product and a fatty alcohol solvent combination of the chlorinated hydrocarbon type, the amounts of petroleum emulsified were, respectively, 2.2, 3.2, 4.0, 6.3 and 8.2 cc.; the half-value times for petroleum were 20, 18, 25, 30 and 28 minutes; the olive oil emulsified was 3.2, 4.2, 3.8, 5.9 and 6.4 cc.; and the half-value times for olive oil were 60, 68, 75, 30 and 25 minutes. Other tests were made under the same experimental conditions to determine the time in which 0.2 g. of oil or fat could be emulsified, yielding an emulsion which was stable for 10 minutes. Using the fatty alcohol-solvent combination of the halogenated hydrocarbon type the last tests yielded a period of 10-12 minutes for bone fat, 15-20 minutes for mineral oils and 18-30 minutes for linseed oils.

Sulfonated (or sulfated) oils of many kinds are very useful as emulsifiers. They vary greatly in their effects because they vary in themselves. Not only do the raw material oils vary in chemical composition but when they are sulfo-

<sup>&</sup>lt;sup>9</sup> Ohl, Spinner U. Weber 52, No. 49, 9 (1934).

<sup>&</sup>lt;sup>10</sup> Sulphated Oils—Burton (1940) Chemical Publishing Co., Brooklyn, N. Y.

nated, the finished products will vary with the concentration of sulfuric acid used, the time, temperature, water content, washing and neutralizing methods, and other related factors.

These sulfonated oils are effective in weakly acid media. They tend to separate out water, hydrolyze on ageing, and split off acid which in turn will react with certain metals with which they come in contact.

In spite of all these disadvantages, sulfonated oils are used in tremendous quantities because they possess certain advantages and are low in price.

They must, however, be bought from reputable houses who standardize their products so that comparable results can be gotten from all shipments.

Analysis of these products is recommended to avoid production difficulties.

The oil most frequently sulfonated is castor. For special purposes nearly all commercial, vegetable, animal and fish oils are used. Some sulfonated oils dissolve clearly in water while others do not. The latter class is preferred in fatliquoring of leather.

Ruemele <sup>11</sup> gives ways of determining the efficiency of the emulsifying power of sulfonated oils.

Sulfonated hydrogenated castor oil is superior to commercial sulfonated castor oil as an emulsifying agent for certain products; a good 25% oil emulsion is produced with 1.25% of the former while an emulsion with 10% of the latter breaks. Sulfonated hydrogenated castor oil is likewise preferred to commercial sulfonated castor oil for production of cosmetics. A stable cold cream is readily prepared with 0.035% of sulfonated hydrogenated castor oil as the sole emulsifying agent;

<sup>&</sup>lt;sup>11</sup> Ruemele, Seifens-Z-62, 393 (1935).

and paraffin oil-water. A similar study was made of egg yolk on the system benzene-water, at 15°. Bile and egg yolk proved to be strong emulsifiers, and in the case of bile, the greater the difference in polarity of the two nonmiscible liquids, the greater its action.

By the interaction of borax and fatty acid, whether cerotic or stearic acid, 18 the resulting sodium soap usually causes an oil-in-water emulsion. When 25 per cent water, 14.5 per cent white wax, about 60 per cent liquid paraffin and not over one per cent borax is used, a water-in-oil emulsion forms, which becomes less stable as the borax content approaches one per cent. The change of phases is not dependent on the white wax; it takes place in precisely the same manner with a mixture of paraffin hydrocarbons.

The antagonistic effect among emulsifiers which individually produce the same type of emulsion, i.e., oil-in-water emulsions or water-in-oil emulsions, often breaks such emulsions, when the emulsifiers are mixed. In the preparation of emulsions of pure Norwegian cod-liver oil for pharmaceutical purposes several interesting cases of antagonism of emulsifiers were observed. Both bile salts and gum acacia, when present alone, produce stable emulsions; but when present together no emulsion forms. With Turkey-red oil, lecithin and sodium oleate, it was found that when mixed with gum acacia, the emulsion formed liberates oil slowly at the top, although when gum acacia is present alone no such separation is observed. Gum acacia, when mixed with gelatin, egg albumin, saponin, Irish moss, agar-agar or gum tragacanth instead of causing liberation of oil, stabilizes the emulsion. Emulsifiers can be classified into 3 groups: (1) emulsifiers with low internal and superficial viscosity like sodium oleate; (2) emulsifiers with low internal and high superficial viscosity

<sup>18</sup> Bushby, Pharm. J. 135, 345 (1935).

like saponin; and (3) emulsifiers with high internal and superficial viscosity like gum acacia. A general conclusion is drawn from the experimental study, that emulsifiers of group (1) antagonize the other groups and cause liberation of the oil at the top. Emulsifiers of groups (2) and (3) do not have mutual antagonistic action. If, however, a large amount of gum acacia is used with group (3) emulsifiers, the emulsions lose consistency, become thin and water quickly separates at the bottom. <sup>18a</sup>

Emulsifiers especially applicable to pharmaceutical use:

- a. Acacia is the most commonly used emulsifying agent for oil-in-water emulsions. Acacia and other gums should not be used in emulsions intended for external application, as they leave a sticky layer on the surface of the body.
- b. Tragacanth is next in importance to acacia but is not as soluble in cold water. If used in conjunction with other good emulsifiers, it produces the viscosity necessary to stabilize the emulsion.
- c. Agar absorbs considerable quantities of water and in this respect is similar to tragacanth. It is useful when combined with acacia and must be used in solution and kept warm until the emulsion is completed, as agar gels at about 40°C.
- d. Gelatin should not be combined with other emulsifying agents. Gelatin solutions are used to some extent for making liquid petrolatum emulsions.
- e. Chondrus mucilage prepared from chondrus is seldom used, except in large-scale operations, because of the time required for preparing it.
- f. Egg yolk is a natural emulsion and may be used in preparing artificial emulsions.

Chatterjee, J. Indian Chem. Soc. 13, 563 (1936).

- g. Soap is an excellent emulsifying agent but is not generally used for preparing emulsions for internal use on account of its laxative action and unpleasant taste. Lime liniment (carron oil) is a water-in-oil emulsion in which the soap formed by the action of the lime water on linseed oil, is the emulsifying agent.
- h. Alkalies are frequently listed as emulsifying agents but they act by saponifying fats and oils, producing soaps which are the actual emulsifiers.<sup>18b</sup>

Pectin produces coarse emulsions of apricot kernel, cod liver and mineral oils and is not a satisfactory emulsifying agent when used alone. When one-half of the acacia is replaced by a small amount of pectin, the emulsion produced is satisfactory, even though microscopic examination shows the globules to be somewhat larger than those produced by acacia alone. 18c.

# Frothing and Protective Action.

Saponin lowers the surface tension <sup>19</sup> of aqueous solutions, being itself concentrated at the interface. This is the cause of the very marked frothing of such solutions when shaken with air. The film of concentrated saponin solution which encloses the bubbles is characterized by a considerable amount of rigidity which accounts for the great stability of the foam, but this is destroyed by the presence of surface-active substances such as alcohol or ether, which displace the saponin from the film.

The extreme dilution at which this frothing may be observed varies with the different saponins, and may be used to characterise them and also as a means of assay of saponin-

<sup>&</sup>lt;sup>18h</sup> Methods for Pharmacy Technicians, p. 117, U. S. War Dept. 1941. <sup>18c</sup> Goldner, J. Amer. Pharm. Ass'n. 114, 44, (1942).

<sup>&</sup>lt;sup>19</sup> The Saponins. G. Middleton, Ind. Chem. Pharm. & Cosmetic supplement May 1935.

containing drugs. The "Foam value" of a number of samples of senega root has been found to vary from 1,000 to 3,300; that of sarsaparilla root from less than 40 up to 660. There does not appear to be any relation between the foam values and the haemolytic index.

The concentration of saponin at the interface also occurs when the second phase is a liquid, e.g., an oil, so that saponins form useful emulsifiers. They have an advantage over soaps and other compounds of a salt-like nature, in that their emulsifying power is not appreciably affected by the alkalinity or acidity of the solution within the limits usually employed.

# Hydrotropic Solvents

Compounds of the type RSO<sub>3</sub>M; where M is sodium, potassium, calcium, lithium, or an ammonium group; R is a long chain paraffin or aromatic group; are hydrotropic solvents.

A concentrated water solution (40% or more) dissolves many water insoluble materials. In some cases, emulsions first form and often may be cleared by the judicious addition of water.

Some hydrotropic solvents are:

Sodium Kerosene Sulfonate Calcium Xylene Sulfonate Calcium Lignin Sulfonate

These have been used in making emulsions and solutions of cellulose acetate, pyroxylin, nitrobenzol, methylsalicylate, anethol, essential and perfume oils.

<sup>20</sup> Kofler, Pharm. Monat. 3, 117 (1922).

#### CHAPTER II

## TYPES OF EMULSIONS AND FOAMS

# Emulsion Types

EMULSION may be oil in water (O/W), water in oil (W/O) or dual emulsions. An O/W emulsion is one whose continuous (outside) phase is aqueous. A W/O emulsion is one whose continuous (outside) phase is oily or water insoluble. A dual type of emulsion is made up of a mixture of the above two types of emulsions.

A number of methods are available for determining the type of emulsion.

Since an emulsion can only be diluted with the continuous phase, it is added to a little water. If it spreads and mixes with the water, it is O/W; if not it is W/O.

If a little oil soluble dye is dusted on the surface of an emulsion, the latter will only be colored if oil is the continuous phase (W/O). If it is not colored, then it is O/W. This of course, presupposes that the dye powder does not invert the emulsion.<sup>1</sup>

If two electrodes are introduced into an emulsion and a current applied, little or no current will flow (as indicated by galvanometer)<sup>2</sup> if the oil phase is continuous. If a flow of current is shown, then the water phase is continuous and the emulsion is O/W.

<sup>&</sup>lt;sup>1</sup> The Elements of Colloidal Chemistry, Freundlich, London, Eng. <sup>2</sup> Pullar, Chemist & Druggist, 127, 742 (1937).

A parallel bundle of light rays, going through a droplet of Q/W emulsion will be converged; through a W/O emulsion it will be diverged.<sup>3</sup>

W/O type emulsions, usually form when the ratio of water to oil is low (less than 1). The addition of more water often reverses the emulsion.

Where a W/O type is desired the water should be added to the oil. For an O/W type the oil should be added to the water. This procedure does not imply that the desired type will always form, only that with all factors equal, it will be inducive to a certain type formation.

Hydrophilic <sup>4</sup> emulsifiers (such as gum arabic, gelatin or soap) <sup>5</sup> tend to form oil in water emulsions. Hydrophobic emulsifiers (such as metallic soap) tend to form water in oil emulsions. Emulsifiers which have both hydrophilic, and hydrophobic position may produce either type. Where one of the phases has a greater affinity for the emulsifier it tends to become the dispersion medium, for then the droplets of the disperse phase can build up a protective sheath from the emulsifier.<sup>8</sup>

Thus, if an oil is shaken with a dilute soap solution, an O/W type results because the soap is soluble in the continuous and not in the disperse phase. If a little calcium oleate is dissolved in the oil (with warming) and water is then mixed in, a W/O emulsion results.

In W/O emulsions, if the oil is lighter than water, it will cream downward; if more dense, upwards. In O/W emulsions creaming directions are the opposite.

The term "dual emulsion" is applied to emulsions of the same pair of liquids, with the same emulsifier, in opposite

<sup>&</sup>lt;sup>3</sup> Carriere, Chem. Weekblad 26, 413 (1929).

<sup>&</sup>lt;sup>4</sup> W. Meyer, Farben-Chemiker 5, 157 (1939).

<sup>&</sup>lt;sup>5</sup> Elements of Colloidal Chemistry, Freundlich. <sup>6</sup> Wellman, J. Phys. Chem. 34, 379 (1930).

types. A dual emulsion is usually made by changing the phase volume ratio. In some cases it is formed by mechanical action. There is also the possibility of chemical reaction between the emulsifier and one or both phases, producing a different emulsifier which tends toward inversion and in certain cases toward duality. Thus, the chemical and physical character of the components is just as important as the kind of emulsifier used. For example, if the only variation in a given system is in the nature of the oil phase the same or different type of emulsion will be formed with varying stability. Thus, while gelatin usually gives O/W emulsions with most oils, with cresol it forms W/O.

Dual type emulsions may be formed by purely physical changes. A protein emulsifier may hydrolyze or be denatured; one of the phases may cause a change in pH; a colloid may assume different states of aggregation and so on.

Partition of the emulsifier between two phases may also produce dual type emulsions. Thus, where an emulsifier is soluble in both phases, the change in amount of either phase will change the specific partition of the emulsifier.

Lecithin dispersions form dual type emulsions with benzene, carbon tetrachloride, chloroform, cresol, hexalin, linseed oil, phenol, and light petroleum. It is quite likely that the partition of the emulsifier, between both phases, is responsible for the duality. Then again, it may be due to the presence of impurities like cephalin.

Hevea lipin also forms dual type emulsions as do proteins and similar substances, such as peptone, albumin, casein and gelatin. Here the emulsifier may form protein complexes with resultant duality of type.

In the system acetic acid, benzene and water, there are three regions, one in which oil-in-water type emulsions result, another in which water-in-oil type emulsions result and an intermediate region in which the type depends on the method of shaking. Foam forms in an oil-in-water type emulsion.

The results of Cheesman <sup>7</sup> throw doubt on the rule that a given emulsifying agent is capable of stabilizing an emulsion of one type only.

Oil-in-water and water-in-oil emulsions were prepared with the pairs water-amyl alcohol and water-kerosene, with the following emulsifying agents: sodium oleate, magnesium oleate, sodium cetylsulfonate; finely divided carbon (pigment black) and bentonite were used with both water-kerosene and water-amyl alcohol, as were oleic acid, triethanolamine, triethanolamine oleate, saponin, castile soap, rosin soap, sulfonated castor oil, gelatin, finely divided iron, finely divided silicon dioxide and calcium caseinate. The emulsions were prepared by three methods and their stability was measured by determining the time required for 5 cc. of the disperse phase to separate from 50 cc. of the emulsion. In most cases, the stability of the unusual form of the emulsion (for a specific emulsifying agent) is of the same order as that of the usual form. In nearly every case the phase volume ratio and the proportion of the emulsifying agent are identical for the oil-in-water and water-in-oil types.

## Inversion

When the amounts of two phases in an emulsion are in a certain proportion an O/W emulsion <sup>8</sup> may be changed to a W/O type. <sup>9</sup> Thus an O/W soap emulsion may be inverted by the addition of calcium or zinc salts, which form metallic soaps that favor the W/O type. This inversion is easily shown by the increase in electrical resistance of the latter.

<sup>&</sup>lt;sup>7</sup> Cheesman, Trans. Faraday Soc. 34, 594 (1938).

<sup>8</sup> Wellman, J. Phys. Chem. 34, 379 (1930).

<sup>&</sup>lt;sup>9</sup> Bhatnagar, J. Chem. Soc. 117, 542 (1920); 119, 61, 1766 (1921).

Inversion may be caused by change in concentration of one of the phases, by chemical or physical changes produced on ageing.

It has been shown in certain emulsions, that when the aqueous soap concentration is increased beyond a certain point some soap is taken up by the other phase. This also occurs when salts are added. In both these cases, as well as in others, where solid or colloidal material is shown in the oil phase, inversion to the W/O results.<sup>10</sup>

Sodium chloride inverts W/O emulsions when the less soluble sodium soaps are used as emulsifiers, but not when the more soluble ones are used. Potassium chloride does not invert emulsions made with corresponding potassium soaps. <sup>11</sup> Cod liver oil emulsions with gum acacia and gum tragacanth change from the less stable O/W to W/O type, during emulsification, caused by slight oxidation of the oil. <sup>12</sup>

W/O crude asphaltic oil emulsions are inverted with potassium or sodium oleate or rosin soap.<sup>18</sup>

Inversion of olive oil emulsions, made with sodium oleate, takes place gradually, not suddenly, in presence of calcium salts.<sup>14</sup>

O/W emulsions of castor, olive, or cod liver oil are formed when using caesium, potassium, sodium, or silver soaps. The degree of emulsification decreasing with atomic volume in above order. Calcium, magnesium, zinc, aluminum, iron hydroxides form W/O emulsions. The stability of these increases inversely with the atomic volume.<sup>15</sup>

Change in type of emulsions of castor or olive oil with

Robertson, Z. Koll. Chem. 7, 7 (1910).
 Tartar, J. Phys. Chem. 34, 378 (1930).
 Smith, Pharm. J. 125, 91 (1930).
 Kadt, J. Inst. Petroleum Tech. 12, 217 (1926).
 Fines, Sci. Deep B. 13, 166 (1930).

 <sup>14</sup> Dixon, Sci., Proc. Roy. Dublin Soc. 19, 421 (1930).
 15 Iselin, Boll. chim. farm. 67, 225 (1928).

sodium oleate is indicated by a change in surface tension of the emulsion 16

Water added to a 4% solution of potassium oleate in white spirit forms a rather unstable water in oil emulsion until the emulsion contains 1% H2O. A further addition of water results in an inversion of the emulsion and a separation of white spirit. The inversion is prevented if the potassium oleate contains more than 10% oleic acid. When water is absorbed by a white spirit solution containing potassium oleate, oleic acid and methanol, one of the three types of emulsions results. If the oleic acid content is high and the methanol content small, a low-viscosity irrevertible emulsion forms. With solutions low in oleic acid and containing between 30% and 40% methanol a water in oil emulsion of higher viscosity forms which has a tendency to invert. If the methanol content exceeds 50%, no emulsion forms.

Olive oil and lime water when emulsified with small amounts of powdered mustard produce W/O emulsions. Larger amounts of mustard produce O/W type. Fine mustard is more effective than coarse mustard. The stability of mayonnaise is definitely affected by mustard.17

In general, emulsions are inverted to W/O if:

- 1. No electric charge exists in the film
- 2. Considerable rigidity exists

Addition of multi-valent ions (or charged molecules containing many polar groups) causes discharge (enabling droplets to coalesce) and greater rigidity of the interfacial film through interlacing of film molecules by the added ion.18

Univalent electrolytes are incapable of reversing the type

<sup>16</sup> Joshi, Kolloid-Z 34, 280 (1924).

Corran, Spice Mill 57, 175 (1934).
 Schulman, Trans. Faraday Soc. 36, 661 (1940).

of soap emulsions, even though they throw the soap out of solution. An excess of adsorbed positive ions on the surface membrane leads to the formation of the water-in-oil type, and negative ions the reverse type of emulsion. Evidence favors the view that the relative wetting power of the two phases with respect to the emulsifying agent and the surface potential of the membrane between them are the main factors governing emulsifications, and that reversal of phases by electrolytes is effected by relevant changes produced in the surface membrane.<sup>17a</sup>

Emulsions of benzene with sodium oleate, and tetrahydronaphthalene with sodium oleate are of the oil-in-water type throughout, and the viscosity decreases continuously with increase in water content. Emulsions of benzene with sulfonated fish oil and caustic soda and tetrahydronaphthalene with sulfonated fish oil and caustic soda are water-in-oil and the viscosity increases with the water content up to 23 and 27% respectively. At these points the systems suddenly change into oil-in-water types and the viscosity increases enormously, after which the viscosity decreases sharply with further increase in water content.<sup>18</sup>

Reversible emulsions (W/O) of castor oil, olive oil and paraffin oil with sodium and potassium soaps increase in viscosity with increase in percentage of the dispersed phase and the viscosity is greatest at the reversal point. The concentration and specific nature of the emulsifying agent have no effect on the viscosity. In pharmacy, a little saponin is often added to gum emulsions to increase their stability.

Saponins also act as protective agents to hydrophil colloids such as the colloidal solutions of the metals, though in gen-

<sup>&</sup>lt;sup>17a</sup> Bhatnagar, J. Chem. Soc. 119, 1760 (1921).

<sup>&</sup>lt;sup>18</sup> Parke, J. Chem. Soc. 1214 (1933).

<sup>19</sup> Joshi, Trans. Faraday Soc. (1925).

eral they are less effective for this purpose than a number of other protective agents.

In using gum arabic <sup>21</sup> for making emulsions, the best amount of gum is the total of the weight of olive oil and twice the weight of the dissolved substance \* in it. It seems that the specific gravity of the oil and gum solutions influence, to a great extent, the stability of emulsions.

#### Lecithin

With olive oil <sup>24</sup> and kerosene, <sup>25</sup> lecithin gives O/W emulsions. It is soluble in various organic solvents <sup>26</sup> and combines with acids and bases. With some non-aqueous fluids, dual type emulsions may be formed because of solute-solvent complexes or by partition of the emulsifier. Such dual emulsions are formed with toluol, benzol, carbon tetrachloride, chloroform, linseed oil, cresylic acid, phenol, hexalin and light petroleum oil. Lecithin gives O/W emulsions <sup>27</sup> with certain bases. Added to soap it increases surface tension and drop number. Foam number is increased and foam volume is decreased.<sup>28</sup>

Impure soyabean lecithin has better emulsifying properties than purified lecithin.<sup>20</sup>

With sunflower oil-in-water emulsions, when the proportion of the phases is not changed, then the most stable emulsions are obtained only with the use of definite amounts of

<sup>&</sup>lt;sup>21</sup> Westerhof, Pharm. Weekblad 76, 811 (1939).

<sup>\*</sup> Dissolved substances are the following: bromoform, chloroform, iodoform, carbon tetrachloride, alcohol, diethyl ether, peppermint oil and fennel oil.

<sup>&</sup>lt;sup>24</sup> J. Phy. Chem. 39, 587,738,834 (1923).

<sup>&</sup>lt;sup>25</sup> J. C. S. 119, 1760 (1921).

<sup>&</sup>lt;sup>26</sup> Biochem. Z. 32, 146 (1931).

<sup>&</sup>lt;sup>27</sup> J. C. S. 51, c. (1932).

<sup>&</sup>lt;sup>28</sup> The Soybean Industry, Horwath. p. 134 (1939) Chemical Publishing Co., Inc.

<sup>&</sup>lt;sup>29</sup> Puchkobskii, Colloid, J. (U. S. S. R.) 3, 643, (1937).

lecithin; <sup>30</sup> when larger or smaller amounts of lecithin are used unstable emulsions are obtained. When the amount of lecithin is constant, the character of the emulsions depends upon the proportion of water and oil. With slight amounts of oil, mainly oil-in-water emulsions are obtained and with small amounts of water emulsions of water in oil are obtained. The most stable emulsions are obtained when the phases are present in the same ratio as used in the commercial production of margarine.

#### Gelatin

Gelatins from which organic and inorganic impurities have been removed are more efficient emulsifying agents than the same gelatins with the impurities.<sup>31</sup> The source from which the gelatin is obtained and the treatment to which it is subjected before the gelatin is extracted, has a marked effect on the emulsifying power of the gelatin. Gelatins which have been previously treated with alkali are more efficient as emulsifiers. Oils containing double or triple linkings are more easily emulsified with alkali treated gelatin than saturated hydrocarbon oils.

In using gelatin as an emulsifier the best results are obtained by (1) lowering the surface tension as much as possible: (2) no excess of strong acids, bases or liquefying salts should be added; (3) the viscosity should be as near that of water as possible. No evidence is shown (in kerosene-water emulsions) that gelatin particles are taken up from the solution to form adhesive layers about the oil droplets.<sup>32</sup>

Variation of pH (of emulsions made with gelatin concentration 0.25–1.5%) between pH 3, 4.7 & 6 show little effect. Changing the pH of a gelatin solution alters its emulsi-

<sup>&</sup>lt;sup>30</sup> Strukova, Chem. Zent. 11, 1460 (1936).

 <sup>&</sup>lt;sup>31</sup> Kernot, J. Soc. Chem. Ind. 47, 96 (1928).
 <sup>32</sup> Tice, Amer. J. Pharm. 1, 2 (1939).

fying properties similarly to the changes in its surface tension and viscosity. Stable emulsions show a concentration of gelatin at the interface.<sup>33</sup>

A homogenizer is preferable to a colloid mill in making liquid emulsions with gelatin as it gives finer and more uniform emulsions. It is important to exclude air or other gases as far as possible.

Of the different varieties of gelatin, bone, calf-skin and pork-skin, the latter gave best results in a typical oil emulsion. It does, however, increase the rate of creaming. Gelatins having a high Bloom rating (25g. or higher) give better emulsions. Of course, the method of preparation of the gelatin, its age, purity and isoelectric point, are very important factors. In solutions, gelatin becomes positively charged as the pH decreases below its iso-electric point.84 Dispersed oil particles bear a negative charge. To further adsorption of gelatin at the oil-water interface, it is necessary to build up the electrical charge at the interface. Using pork-skin gelatin of a good grade, pH 3 was found most suitable. Since good commercial gelatins have a higher pH, the latter is decreased by the addition of acid (tartaric acid is used for pharmaceutical emulsions). Alcohol, sugar and glycerin are compatible with gelatin emulsions. The latter two, in certain quantities, improve the emulsion. Emulsions made with hot gelatin solutions are more stable than when cold gelatin solution is used.

Gelatin solutions hydrolyze on warming and on ageing. Hydrolysis produces less viscous emulsions but does not hurt stability very much.

An alkali treated high grade gelatin (Pharmagel B) of isoelectric point 4.7 requires a much higher pH (around 8)

<sup>&</sup>lt;sup>33</sup> Ahukov, J. Russ. Phys. Chem. Soc. 59, 1061 (1927). e, J. Amer. Pharm. Assn. 24, 1063 (1935).

for making good emulsions. The pH is adjusted with sodium bicarbonate.

Maximum stability of benzene emulsions with gelatin is at the latter's iso-electric point. Non-hydrated and undissociated gelatin molecules serve as best emulsifying agents.<sup>33</sup>

Gums such as acacia, tragacanth and agar show a negative charge formed between the oppositely charged colloids.<sup>36</sup>

Where casein is treated with an aqueous alkali the latter reacts with some of the acidic components to form an emulsifier which disperses the balance of the casein. The greater the amount of certain alkalies used, the more nearly transparent the dispersion becomes.

Albumen is positively charged in acid solution and negatively charged in alkaline solution.<sup>35</sup>

# Soaps

Soaps used most frequently as emulsifiers are the sodium or potassium salts of lauric, oleic, palmitic and stearic acids or mixtures of these. They are easily hydrated (solvable), foam in water, lower surface tension and increase detergency. They ionize like salts in water, but show only half the osmotic pressure of the latter. As the temperature of an aqueous soap solution increases the particle size tends to change from colloidal to molecular. Thus, soap solutions are composed of colloidal particles, colloidal ionic micellae and simple ions. Hydrolysis is only slight and any free fatty acid recombines to form an acid soap.<sup>37</sup> Acid soaps are sometimes better emulsifiers than neutral or alkaline soaps. Thus, a dilute potassium laurate that would not produce a

<sup>&</sup>lt;sup>33</sup> Ahukov, J. Russ. Phys. Chem. Soc. 59, 1061 (1927).

<sup>36</sup> De Jong, Biochem. Z-(1929).

<sup>35</sup> Hofmeister's Beitr. Chem. Physiol. 5-27, (1904); Pfluger's Archiv. 24, 247 (1887).

<sup>37</sup> Alexander, Colloid Chemistry 1, 137 (1926).

good emulsion with benzene is considerably improved by the addition of a little lauric acid.<sup>38</sup>

Soaps will take up considerable amounts of water, the amounts increasing with the molecular weight of the fatty acid radical. Thus, 1 gram molecule of sodium arachidate will hold 37 liters water.<sup>39</sup> Alcohol and other solvents are taken up similarly.

Emulsifying power of sodium soaps increase up to N/300 soap solution, above which there is a decrease due to the coagulating effect of sodium ions.<sup>40</sup> The optimum amount of soap for benzene emulsions is 0.01% of the total volume.<sup>41</sup>

Potassium soaps are preferable to sodium and ammonium soaps. The latter decompose and liberate free fatty acid which, is often objectionable.

Naphthenic acid soaps <sup>48</sup> are suitable for the preparation of emulsions, but particular care must be taken in determining the amount of alkali necessary for saponification; excess of sodium hydroxide or potassium hydroxide causes rapid disintegration of the emulsion, whereas in saponification with ammonia an excess (up to twice the theoretical amount) must be present in order to stabilize the emulsion. The sodium salt of naphthenic acid is less easily hydrolyzed in water than are the common sodium soaps and its emulsifying powers are much better.

In making emulsions with triethanolamine soaps, quick thorough agitation is necessary. If the agitation is too vigorous or too prolonged, the emulsion tends to break.<sup>44</sup>

Stable W/O emulsions (with metallic soaps) 45 are best

<sup>&</sup>lt;sup>88</sup> McBain, J. Amer. Chem. Soc. 54, 3266 (1932).

<sup>89</sup> Fisher, Soaps & Proteins.

<sup>40</sup> Donnan, Kolloid-Z 1, 208 (1910)...

<sup>&</sup>lt;sup>41</sup> Briggs, J. Phys. Chem. 19, 479 (1915).

<sup>43</sup> Ohl, Monatsh. Seide Kuntseide 43, 235 (1938).

Freeman, J. Am. Pharm. Assoc. 22, 232 (1933).
 Pink, Trans. Faraday Soc. 37 180 (1941).

when the soap does not dissolve in either phase and remains at the interface being partially wetted by both phases, and when there is sufficient lateral adhesion between the soap particles.

Variation of the hydro-carbon chain of soaps has little effect upon distribution of particles in an emulsion, except in the case of potassium chaulmoograte.<sup>46</sup>

The emulsifying power of soaps toward the system water-benzene is in decreasing order castor oil soap, sodium oleate, coconut soap.<sup>47</sup> This is exactly the reverse of the series for foam formation. By the method of phase reversal, it is shown that the hydrophilic order of the soaps is the same as the order of their emulsifying power. In the presence of calcium ion coconut soap gives the most stable water-in-oil emulsion at a concentration of 0.05% and castor oil soap at 0.01%. Kaolin particles less than ten microns in diameter also stabilize water-in-oil emulsions and therefore are hydrophilic.

Partial polyhydric esters of higher fatty acids. 48a The only polyhydric alcohol fatty acid esters occurring naturally are the triglycerides which are present in various animal, vegetable and fish oils and fats.

Fatty acid esters of polyhydroxy alcohols having a higher molecular weight than glycol and glycerol, as well as esters or glycol ethers, have recently been made available commercially. These are exemplified by the fatty acid esters of mannitol, sorbitol, glycol ethers, polymerized glycols and glycerin.

The glyceryl esters of stearic acid will serve to illustrate the structural formulae of these materials:

Harkins, J. Am. Chem. Soc. 51, 1674 (1929).
 Smirnov, Vs. Na. Is. Inst. Zhirov 116 (1935).

<sup>48</sup>a Catalog Glyco Products Co. Inc., Brooklyn, N. Y.

GLYCERYL MONOSTEARATE	GLYCERYL DISTEARATE
Ο	Ο
"	. ""
H <sub>2</sub> C-O-C-C <sub>17</sub> H <sub>35</sub>	$H_2C-O-C-C_{17}H_{35}$
1	1
HC-OH	HC- $O$ - $CO$ - $C$ <sub>17</sub> $H$ <sub>35</sub>
1	1
H <sub>2</sub> C-OH	H̃₂C-OH

It must, however, be borne in mind that the usual commercial products are not single chemical entities but are mixtures of the mono fatty acid esters, the di-fatty acid esters and, in the case of the glyceryl and hexahydric compounds, small percentages of the tri-esters with perhaps a percentage of free fatty acid. Furthermore, as these are poor emulsifying agents, the addition of small amounts of soap, wetting agent or other surface-tension reducing agents is sometimes necessary to give them emulsifying and wetting properties. It is interesting to note that the amount of surface tension reducing agent necessary is extremely small, the resultant emulsifying power being greatly in excess of that of the ester and soap separately.

Liquid esters of the emulsifying type are being used as emulsifying agents in the manufacture of fluid emulsions in textiles, paper, leather, polishes, cosmetics, and certain types of edible emulsions.

The solid esters of the emulsifying type give stable cream or paste emulsions for numerous industries. They are also of interest as suspending agents in aqueous media, lubricants, shortenings and other edible products.

Typical emulsifiers in this group are:

Glyceryl (Mono) Ricinolate Glyceryl (Mono) Stearate Mannitol Glyceryl (Mono) Laurate Mannitol Glyceryl (Mono) Oleate

Mannitol (Tri) Ricinoleate Mannitol Glyceryl (Mono) Stearate Polymerized Glycol (Di) Laurate Polymerized Glycol (Mono) Laurate Polymerized Glycol (Di) Oleate Polymerized Glycol (Mono) Oleate Polymerized Glycol (Di) Stearate Polymerized Glycol (Mono) Stearate Propylene Glycol (Mono) Laurate Propylene Glycol (Mono) Oleate Propylene Glycol (Mono) Ricinoleate Propylene Glycol (Mono) Stearate Propylene Glycol Glyceryl (Mono) Stearate Sorbitol Laurate Sorbitol (Di) Laurate Sorbitol (Di) Stearate Sorbitol Glyceryl (Mono) Laurate Sorbitol Glyceryl (Mono) Oleate Sorbitol Glyceryl (Mono) Stearate Diethylene Glycol (Mono) Laurate Diethylene Glycol (Mono) Myristate Diethylene Glycol (Mono) Oleate Diethylene Glycol Glyceryl (Mono) Oleate Diethylene Glycol (Mono) Palmitate Diethylene Glycol Palmitate Stearate Diethylene Glycol (Mono) Ricinoleate Diethylene Glycol Glyceryl (Mono) Ricinoleate Diethylene Glycol Stearate Diethylene Glycol (Mono) Stearate Diethylene Glycol Glyceryl (Mono) Stearate Ethylene Glycol (Mono) Laurate Ethylene Glycol (Mono) Oleate Ethylene Glycol (Mono) Ricinoleate Ethylene Glycol (Mono) Stearate Glyceryl (Mono) Laurate Glyceryl (Mono) Myristate Glyceryl (Mono) Oleate

Glyceryl (Mono) Palmitate

### Twitchell Reagent

The emulsifying power of Twitchell reagent <sup>48</sup> on olive oil is increased by acid. The stronger the acid, the greater the effect.

Addition of salts of weak acids has but little influence on such emulsions. Strong electrolytes (sodium chloride) increase emulsification. Higher concentrations of salt, salt out the emulsifier and break the emulsion.

The emulsifying power of the sodium salts of the reagent is lower than that of the reagent. Emulsifying power is increased here by certain amounts of glycerol or fatty acids.

### Insoluble Emulsifying Agents.

The most useful emulsifying agent in this class is bentonite. While many solids have been used as emulsifiers most of them give coarse temporary emulsions. Hydrous oxides and hydroxides of metals are most effective, their emulsifying efficiency varying with the method of precipitation. The most gelatinous, highly dispersed products are usually best; gelatinous precipitates other than oxides and hydroxides are also effective. Ageing in some cases helps and in other cases hinders their emulsifying power.

Aged aluminum hydroxide and partially coagulated ferric hydroxide solutions are better than freshly precipated materials.

Nearly all colloidal hydroxides give O/W emulsions.

Emulsions made with gelatinous hydroxides although rather coarse are quite stable and are not effected by electrolytes. Finer dispersions are obtained by combining surfaceactive materials with them.<sup>50</sup>

The following is a list of some insoluble emulsifying agents

<sup>&</sup>lt;sup>48</sup> Nishizawa, Chem. Unscha Fette *36*, 277, (1929). <sup>50</sup> Bennistee, J. Soc. Chem. Ind. *59*, 226 (1941).

used; moist basic metallic sulfates,<sup>51</sup> plaster of Paris, calcium carbonate, powdered coals. The finer the particle size of the emulsifier the finer the emulsified droplets. When O/W emulsions are to be formed the emulsifier should be preferentially wetted by water. Other insoluble emulsifiers are arsenic trisulfide; <sup>52</sup> zinc and iron dust, yeast; <sup>53</sup> hydroxides of zinc, lead and aluminum <sup>54</sup> (when pure and in low concentration these give W/O and O/W on addition of electrolytes); carbon black and lampblack gives W/O <sup>55</sup> with kerosene; a very stable emulsion of gasoline is prepared with carbon black.<sup>56</sup> The greater the concentration of insoluble emulsifier the finer the droplet size.<sup>57</sup>

Both W/O and O/W emulsions have been made <sup>58</sup> with bentonite, lead sulfate emulsifies nitrobenzol in mineral acids, <sup>59</sup> kieselguhr, <sup>60</sup> and bauxite. <sup>61</sup>

The following technical aspects of emulsification are discussed in detail by Langton: <sup>62</sup> fatty oil saponification, manufacture of margarine, compound fats, lubricants, asphalt and bitumen emulsions.

The amount of emulsifier used 63 is different for each specific emulsifier. Thus 0.5% gelatin may form a stable emulsion whereas attainment of the same stability may require the use of 4% lecithin. The amount of emulsifier necessary is usually greater than that required to form a uni-

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<sup>51</sup> Pickering, J. C. S. 91, 2001 (1907).
<sup>52</sup> Briggs, Ind. & Eng. Chem. 13, 1008 (1921).
<sup>53</sup> Bhatnagar, J. C. S. 119, 1760 (1921).
<sup>54</sup> Bechold, Kolloid Z. 28, 6 (1921).
<sup>55</sup> Schloepfer, J. C. S. 113, 522 (1918).
<sup>56</sup> Odell, U. S. Bureau of Mines Technical Paper 304, 36 (1923).
<sup>57</sup> Moore, J. Amer. Ch. Soc. 41, 940 (1919).
<sup>58</sup> Western, Chem. Age 4, 604, 638 (1921).
<sup>59</sup> Sheppard, J. Phy. Chem. 23, 634 (1919).
<sup>60</sup> Lapworth, J. C. S. 119, 765 (1921).
<sup>61</sup> King, J. S. C. I. 57, 431 (1938).
<sup>62</sup> Langton, Ind. Chemist 3, 457 (1927).
<sup>63</sup> Brown, Textile Colorist 57, 516 (1935).
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molecular layer, especially if hydration occurs. The excess may be in the continuous phase or it may be adsorbed on the covered globules of the disperse phase. This tends to increase viscosity and augment stability. The greater the amount of emulsifier (within certain limits) the smaller the particle size of the emulsion. Therefore, the smaller the particle size desired, the greater amount of emulsifier necessary. Where an emulsion is made with a minimal amount of emulsifier there is a tendency toward liberation of the oil phase and creaming or breaking.

#### Foams

Pure liquids do not foam. 18a Some impurity must be present in a liquid to make it foam. Expressed chemically, a foreign substance must be molecularly or colloidally dispersed in a liquid in order to make foam. The nature of the added substance has little qualitative effect—that is, nearly all substances induce foaming. In a quantitative sense, however, there are great differences. A few thousandths of one per cent of some substances produces more foam than several per cent of others, and there is, furthermore, great difference in the stability of the different foams.

Qualitatively speaking, it does not matter whether the added substance is positively or negatively absorbed—that is, is more or less concentrated in the surface than in the interior of the liquid. In either case foam may be produced. Since positively adsorbed substances lower the surface tension and negatively adsorbed substances raise it, it is apparent that there is a correlation between foaminess and surface tension

Certain substances called antifoams, have the power of destroying foam or preventing the formation of foams pro-

a Foulk, Ind. & Eng. Chem. 21, 815 (1929).

duced by positively adsorbed or negatively adsorbed substances. Castor oil, for example, destroys the foam produced by the positively adsorbed saponin and also that caused by the negatively adsorbed sodium sulfate. Quantitatively considered, the defoaming agents, like the foam producers, are at times effective in concentrations of only a few thousandths of a per cent.

The fundamental condition for the formation of a foam requires that there be a difference of concentration of the dissolved substance in the surface layer, as compared to the concentration of the substance in the bulk of the solution.

If the tension in the film is increased by increasing the area. the concentration of the wetting agent or surface active compound is reduced, thus balancing the increase in tension. Conversely, if the tension of the film is reduced by reducing the area of the concentration the wetting agent is increased and surface tension will be decreased to balance the reduced tension. If a material is too soluble, or too insoluble, it will not produce a copious foam. (A 0.2% concentration of the dioctyl ester of sulphonated dicarboxylic acid forms more foam and a more stable foam than a 0.2% concentration of the dibutyl ester of sulphonated dicarboxylic acid.) Likewise, a 0.2% solution of a sulphated higher alcohol, produces more of a froth and a more stable froth than either of the other two materials. This is in line with experience that detergent-type surface active products are generally better foamers than wetting agent types.8a

The most commonly used foam-breakers, outside of those used in the natural petroleum industry are:

Higher fatty alcohols, e.g., cetyl alcohol Diglycol laurate Animonium linoleate Alcohol, denatured Octyl alcohol

<sup>8</sup>a Sluhan, Paper Trade Jol. p. 1, Aug., 22, 1940.

Pine oil Mineral oil "Foamex" <sup>8b</sup> Sulfonated oils Vegetable oils Water soluble salts, e.g., sodium sulfate Acids, e.g., acetic acid Tallow

Mechanical methods such as beating or stirring near the surface of a foaming liquid; the use of screens, a blast of air, and similar devices are frequently used.

Hydrocarbons, carbon tetrachloride, chloroform and carbon disulphide have little or no effect 9 on the formation of foams. Organic acids, alcohols, amines, aldehydes, ketones, glycerol, sugar, although an aid in the production of foams decrease their stability. The optimal concentration in 35 such compounds for a maximum of foam is given by Bartsch. Surface activity, solubility, degree of dispersion and viscosity are factors in the effectiveness of each substance. In all dilute solutions the foam-forming power increases with increasing surface activity. In concentrated solutions, the influence of dispersion, solubility and viscosity subdue that of surface activity. The surface tension optimal for foam-building, of many solutions, approaches a mean value so that one may speak of the isocapillarity of these solutions. Molecular association or colloidal aggregation decreases the foam-building power of surface-active substances. High viscosity may not aid in the formation of foam, but increases its stability. Mixed solutions of surface-active substances have greater foam-building power than the solution of just one component, unless mixing lowers the solubility. Under the influence of electrolytes, foams usually behave as negatively charged lyophobes. Cations exert a flocculating effect according to their valence, and protective colloids protect in accordance with their gold number. The ability to form foam is con-

<sup>8</sup>b Catalog, Glyco Products Co., Inc., Brooklyn, N. Y.

<sup>9</sup> O. Bartsch, Kolloidchem. Beihefte, 20, 1 (1924).

ditioned by the heterogeneity of the Gibbs layer with regard to the component molecules. The characteristics of a three-phase foam system is primarily dependent on the concentration of the foam builder or stabilizer. There is no sharp division between the three-phase emulsion and three-phase foam. Protective colloids may hinder the formation of foam. The maximum stability is reached when the adsorbed layer of fatty acid is one molecule thick on the solid phase.

Egg-white foam 10 is an emulsion of air in liquid, in which there is a concentration of the coagulated protein at the airliquid interface, which forms a tough skin about 1 molecule thick, thus permitting the emulsion to be semi-stable. The lighter the foam, the less the wall thickness, and there is a point at which the two surfaces are connected by what appears to be a long chain of albumin molecules (possibly coagulated). This gives to the foam the quality of dryness. Egg white is composed of microscopic fibers of protein which make the viscosity very high. On beating, these fibers are cut and torn and a relatively low viscosity results. This allows for the production of a highly dispersed foam. The stability of the foam is a linear function of the viscosity of the egg white drained from it. When certain acids or salts are added to egg white thereby decreasing its pH, a change is produced which is partly due to an increase in viscosity and partly due to some change in the nature of the protein. The result is a more stable foam.

A heterogeneous system consisting of acetic acid-diethyl ether-water acts both foamy and non-foamy <sup>11</sup> according to the different methods of shaking. This behavior is dependent on the type of emulsions produced. Accordingly the heterogeneous region of this system is divided into three parts,

Barmore, Colo. Agr. Exp. Sta. 46th Ann. Report 15 (1933).
 Sasaki, Bull. Chem. Soc. Japan 1r, 63 (1939).

namely, oil-in-water region, water-in-oil region and phase-inversion zone. In the latter, both types of emulsion may be produced by different technics of shaking.

A study of salts of triethanolamine <sup>12</sup> indicates that the surface tension decreases and the relative foam production increases with the number of carbon atoms of the saturated fatty acid up to 12, and then decreases. The pH increases with the number of carbon atoms. The stearate, oleate and laurate exhibit the best emulsifying powers.

Spent licorice root <sup>18</sup> is extracted under pressure with 5% caustic soda, and is then concentrated. This acts as a foam stabilizer, giving a tough durable film.

Foamapin <sup>14</sup> a hydrolyzed protein, gives an excellent tenacious foam film in water. Its durability can be further increased by the addition of inorganic salts.

Stability of whey foams may be greatly increased by addition of (1) small quantities of calcium hydroxide <sup>15</sup> and its subsequent neutralization; (2) successive small quantities of acid and alkali to the foam while whipping; (3) small amounts of tannic acid, saponin, sodium sulfite or bisulfite. If, in addition to any of the above, the whey solution is heated to 50°C. before whipping, volume and stability of foam is increased.

The following materials are listed according to the descending order of their foaming power in hydrophilic colloids. <sup>16</sup> Saponin, molasses, peptone, gelatin, albumin, pectin, casein. For albumin the maximum foaming power occurs just below the coagulation temperature, and maximal foam stability is

<sup>&</sup>lt;sup>12</sup> Fiero, J. Am. Pharm. Assoc. 27, 658 (1938).

<sup>&</sup>lt;sup>13</sup> Houseman, J. Ind. Eng. Che., 21, 917 (1929).

 <sup>14</sup> Glyco Products Co., Inc., Brooklyn, N. Y.
 15 Peter, J. Ind. & Eng. Chem. 22, 1124 (1930).

<sup>16</sup> Dumanskii, Schr. z. biochem. Forsch inst. Nahr. (U.S.S.R.) 3, 361 (1933).

found at pH 4.7. Replacing air by carbon dioxide or sulfur dioxide decreases the stability but not the amount of the foam.

Soaps foam in following order: 17

Castile soap Palm Oil soap Tallow soap Solidpotash soap Palm kernel oil soap Rosin soap

The foaming quality of soaps in hard water, increases when the solution stands. Colloidal substances do not help foaming of soaps or increase the life of the foam.

Clay lowers foaming quality of soaps.

0.001-0.5% of montan wax or I.G. waxes are dissolved in a soluble oil 18 to suppress foam when mixed with water.

Uniform pressure does not destroy foam.19 Unilateral pressure does destroy foam by strong deformation of the bubbles.

<sup>&</sup>lt;sup>17</sup> Anon. Soap *10*, 19 (1926). <sup>18</sup> Zimmer, U. S. 2, 230, 556 (1941).

<sup>19</sup> Siehr, Kolloid-Z. 78, 156 (1937).

### CHAPTER III

# METHODS, FORMULATION, EQUIPMENT

#### Methods

While there are numerous ways of making emulsions the simplest is to add the oil phase slowly, with stirring, to a concentrated water solution of soap. Further amounts of water may be stirred in.

It is often advantageous to add the internal phase to the external phase slowly; to use a preformed dilute emulsion; <sup>1</sup> a preformed concentrated emulsion; <sup>2</sup> to use a preformed emulsion using a different emulsifier than that used in the main emulsion; <sup>4</sup> to remove part of finished emulsion and add an unemulsified phase. <sup>5</sup> 6

Beating in of too much air causes excessive froth formation which is detrimental. Dilution of a concentrated emulsion should be slow to avoid complications.<sup>7</sup>

For emulsions of O/W type it is usual to put the emulsifying agent in the water phase. When soap is used it may be dissolved in the water phase or it may be formed *in situ* by

<sup>&</sup>lt;sup>1</sup> Brit. 4,505 (1912).

<sup>&</sup>lt;sup>2</sup> Lechler, Brit. 254, 701 (1925).

<sup>&</sup>lt;sup>3</sup> Billinghame, U. S. 1,700,581 (1929).

<sup>4</sup> Braun, Brit. 254,012 (1925).

<sup>&</sup>lt;sup>5</sup> Robinson, U. S. 1,923,888 (1933).

<sup>&</sup>lt;sup>6</sup> Neveu, U. S. 1,525,409 (1925).

<sup>&</sup>lt;sup>7</sup> Brown, Textile Colorist 57, 516 (1935).

dissolving the alkali in the water and the fatty acid in the oil phase. The latter method often gives better dispersions because the emulsion forms spontaneously as soon as the two phases meet. Furthermore, less power is necessary for mixing in such a case. Certain emulsifiers which are dispersible in both water and oils (e.g., diglycol laurate) may be dissolved in either the water or oil phase or both, depending on results desired.

The effect of concentration of emulsifiers (albumin, sodium caseinate and casein lactate) and of stirring speed on emulsions is very important.

In making oil emulsions with soap, the oil and water is alternately mixed into the concentrated aqueous soap solution. When a wax is being emulsified, it is usual to run the melted wax into the hot soap solution containing all the water. When emulsifying mineral oil with soaps that are soluble in it, a solution of the two is made and then stirred into the water.

Mineral oil emulsions, however, can be made without an emulsifier, by shaking a small amount of oil with a large amount of water for 48 hours; by boiling a similar ratio under reflux for 30 hours; by dissolving a similar ratio in alcohol and pouring into water.<sup>8</sup>

For petroleum solvents the alkali is dissolved in water and the fatty acid in the solvent. On mixing these solutions soap is formed spontaneously and often gives a finer dispersion than other methods of mixing.

In making an emulsion of a new oil, work the oil gradually into a concentrated soap solution. If the oil does not emulsify well, add water, a little at a time, working it in well. A gel or paste usually forms. Here more oil can be stirred in. If there is incomplete emulsification add water again. This

<sup>8</sup> Lewis, Kolloid-Z 4, 211 (1909).

procedure is not required in all cases, but is often very helpful when an emulsion is made for the first time. In making duplications the entire amount of oil or water may be stirred in more rapidly.

Gums are used as emulsifiers in the following ways: 9

The Continental method: Oil is ground to a paste with powdered gum acacia. The water is added in two parts, the first part being worked into the paste, before adding the second part.

The English method: A mucilage of gum acacia and water is formed and the oil and water is worked in, in successive small portions.

Gum emulsifiers should not be thinned down too much with water as instability will result.<sup>10</sup>

Two immiscible liquids can be emulsified with the help of an insoluble powder, such as clay, zinc dust, or yeast. The emulsification depends on the fineness of the powder, on the quantity of the solid phase, and on the presence of certain substances which are soluble in one or both liquid phases and which influence the wetting of the powder by both phases. These substances include acids, bases and salts, both electrolytic and non-electrolytic. Geometrical considerations have shown that for complete emulsification the optimal ratio of inner to outer phase should be 74—to 26; experimental results show the optimum ratio to be 60 to 80 volumes of the inner to 20 to 40 volumes of the outer phase. The volume of the emulsion is influenced by the specific weights of the solid and liquid phases; great differences in specific weight decrease the emulsion volume.

Bentonite, the most widely used insoluble powder is dispersed properly by adding it to water, a little at a time, and

<sup>&</sup>lt;sup>9</sup> Serralach, J. Ind. & Eng. Chem. 25, 817 (1933).

Roon, J. Ind. & Eng. Chem. 9, 156 (1917).
 Bechhold, Kolloid-Z 28, 6 (1921).

allowing it to sink before adding the next portion. When all the bentonite has been added, allow it to soak for one or two hours, before stirring. Up to 7 or 8% bentonite may be dispersed in this way.

If a high speed mixer is available, the bentonite can be added slowly while mixing, and dispersion can be effected in a few minutes. This method and longer mixing may yield dispersion up to 17%.

## Emulsification by Ultrasonic Waves

Ultrasonic waves <sup>12</sup> are a source of strong mechanical energy which greatly accelerates the speed of molecules within a limited area. Such a source of energy rapidly disperses mercury in water. The proportion of the phases is a critical factor in these entilsions, which are only stable for a day.

More stable emulsions have been obtained with Wood's metal in certain organic liquid-like brombenzene. The stability is further increased by the presence of highly charged and strongly adsorbed ions.

Increasing the disperse phase, beyond a certain point, is not possible even if the treatment time is greatly extended. If treatment of a good emulsion is continued too long, breaking often results. The inclusion of a protective colloid, however, may prevent this. Highly viscous or elastic materials do not emulsify on ultrasonic treatment.

Emulsification in oil-water systems by ultrasonic waves <sup>13</sup> is due to cavitation, emulsification occurring when the cavities collapse. With constant frequency vibration the emulsification increases over a range of 100 to 1500 mm. of mercury pressure, but decreases above this point. Some pressure is necessary to cause efficient collapse, but too high a

<sup>12</sup> Science Progress 23, 1, 75 (1928).

<sup>13</sup> Bondy, Trans. Faraday Soc. 31, 835 (1935).

pressure causes the dissolved gases to buffer the collapse or else does not favor the formation of nuclei. Some dissolved gas is necessary for efficient emulsification, for it aids in the formation of nuclei for cavitation.

# Emulsifying High Melting Materials

When an emulsion of a solid melting above 100°C. is desired, it should first be melted with sufficient solvent or oil to reduce the combined melting point below 100°C. For example, rubber with naphtha or other hydrocarbons; synthetic resins with hydrocarbons or vegetable oils.

### Formulation

The first factor in formulation is the ratio of the oil to the water phase. Usually, an increase in amount of the water phase will result in a lowered viscosity of the emulsion. Often, the decreasing of the amount of the water phase may cause inversion. Low percentages of water in many emulsions produce pasty or even solid emulsions. Since increased viscosity usually increases stability, viscous (concentrated) emulsions are frequently made so that aging and mechanical shock will not break them. These are diluted as required before use.

The amount of the emulsifier used is dependent on many factors, viz., stability, viscosity, type, color, costs, and so forth. It is usual to start out with an amount equal to that used in similar emulsions and work up or down depending on desired results. It is best to work with parts by weight for all ingredients in order to avoid errors due to differences in densities.

Since a test of stability may require "shelf-testing" for some length of time it is best to make up a number of experimental emulsions varying the components (within certain limits) and allowing them to age.

# Variations due to formulation or method

Tars and asphalt emulsions made with caustic soda depend on and vary with the following factors: 1

(1) The type of emulsion depends on the order of introduction of phases, the percentage of phase, and the percentage of potassium hydroxide content in emulsions; (2) the particle size of the dispersed phase varies within close limits; (3) the color of the emulsion depends on that of the prime materials used; (4) the stability of the emulsions depends on type, percentage of tar or mixture of tar-asphalt and viscosity; (5) the viscosity increases with increase in added phase, regardless of whether it is tar or water.

Mechanical forces are instrumental in producing most emulsions. The simplest equipment is a small container which is shaken manually. A second method is stirring, either by hand or mechanically. Finally, there is the machine, such as the colloid mill or homogenizer.

In pharmacy, the mortar and pestle is the most commonly used emulsifying apparatus. The so called American method introduces the oil phase to the gum dispersion, whereas in the Continental method the water is added to the gum and oil. Both these methods use vigorous agitation and grinding.

Standardized hand shaking of a bottle or closed container is the technic which most closely duplicates mechanical forces and is used in many studies on making and breaking emulsions.<sup>2</sup>

Mixing by agitation is not a simple, uniform operation. The shaft, from the source of power, may be fitted with a propeller, whisk, baffle or similar projection. To get the most efficient dispersion, shearing as well as mixing forces are needed. The size, shape and position of both the container

<sup>&</sup>lt;sup>1</sup> Verona, Bul. Chim. Soc. Rom. 38, 91 (1935). <sup>2</sup> Clayton, Theory of Emulsions (1928).

and the attachments are of great importance, not only in getting better dispersions, but in avoiding the introduction of excessive amounts of air.

For every emulsifying apparatus or machine there exists an optimal speed of agitation or mixing and an optimal operating time, whereby the most stable emulsion can be attained for a given mixture. Intermittent shaking may be 600 or even 1000 times more effective than uninterrupted but equally violent agitation. A mixture of benzene in 1 per cent aqueous sodium oleate, when shaken by hand, requires 750 shakes occupying 4.2 minutes, to emulsify 60 per cent by volume of benzene. The same mixture can be completely emulsified with 5 shakes in less than 1 minute, if after shaking twice a rest of 30 seconds is permitted.

This example is mentioned to illustrate the point that results are often unpredictable and are best ascertained by experiment.

Most emulsions are made by means of electric mixers of various types. It is important to select the right type and size of mixer. The number of propellers on the shaft, their location, angle and pitch are likewise often critical.

To get uniform dispersions it is important that the pressure remains fairly constant. Since the pressure, at which the emulsion is brought to the homogenizing valve, may vary, it is important that the valves be quickly responsive to slight pressure variations. The higher the pressure used, the finer is the dispersion. The pressure however, is definitely limited by the size of the inlet valves and the viscosity of the emulsion. Formation of vacuum and air pockets in the compression chamber causes lowered production and variable finished emulsions.

Pressures used vary between 1000 and 5000 lb, sq. in.

Often two stages of homogenization are used; the first at high pressure and the second at a lower pressure. Such a

procedure is used with milk to prevent creaming and excessive viscosity.

# Number of Propellers \*

Generally mixers with one propeller are used with liquids having low viscosity, such as water solutions, oils, light syrups, varnishes, milks, etc.

Two propellers on the mixer shaft are called for with liquids of high viscosity where one propeller cannot set up circulation throughout the entire mass of liquid; also where the liquid in the tank varies in height from time to time.

Note mixing action of single propeller, Fig. No. 1; double propeller, Fig. No. 2. Remember that when two propellers are placed on the mixer shaft, where one propeller requires a certain horse power, each of the two propellers substituted will require approximately half of the horse power as shown by the one large propeller.

## Mixer Speed

A high speed mixer is called for with low viscosity liquids, such as water solutions, light syrups, light oils, varnishes, etc., while low speed mixers are used in high viscosity work where mixes are thick, slippery, etc. Where mix contains crystals, fruit or other solid particles, or tends to foam, low speed units are designated.

Materials containing colloidal matter, such as milk or cream, also require low speeds.

There are also materials requiring medium speeds, such as heavy oils and paints. By high speed we mean 1400 to 1800 R.P.M.; medium 1150 R.P.M.; low speeds 100 to 500, generally around 400 R.P.M.

Wherever possible it is suggested that you use a high speed

<sup>\*</sup>Courtesy of International Engineering Inc., Dayton, O.

Fig. No. 1

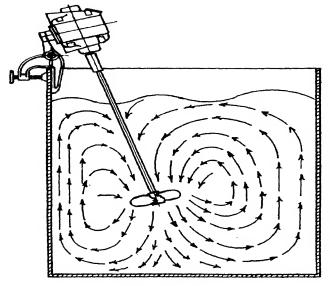
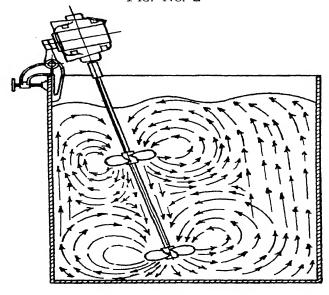


Fig. No. 2



job, as its original cost is less, and it can be handled easier because of its lighter weight.

# Horse Power Required

The horse power required to mix a certain definite quantity of liquid depends entirely upon the viscosity of the liquid. As an example, a cosmetic cream will require 3 to 4 times the power per gallon as compared with an aqueous solution, while rapid agitation of an aqueous solution will take 3 or 4 times the power per gallon as compared with slow agitation.

The following table will give you the approximate horse powers where working on average materials for various capacity tanks, and an approximate comparison of thin or low viscosity liquids with the other extreme, thick high viscosity material, such as pastes, creams and mayonnaise.

### APPROXIMATE AVERAGE HORSEPOWER

	SLOW AGITATION AQUEOUS		RAPID AGITATION AQUEOUS	
GALLONS	SOLUTION	PASTE	SOLUTION	PASTE
15 to 20	1/30	1/4	1/8	1/2
30 to 40	$\frac{1}{2}$ 0	1/3	1/4	3/4
60 to 90	1/20	1/2	1/3	1
150	1/8	3/4	1/2	11/2
250-350	1/4	1	3/1	2
500	1/3	11/2	1	3
800	1/2	2	1 1/2	5
1200	3/4	3	2	71/2
2000	1	5	3	10
3000	1 ;	71/2	5	
5000	2	10	71/2	
<i>7</i> 500	3		10	

NOTE: Medium agitation can be figured to average approximately 2/3 of the above Rapid Agitation.

#### Miscellaneous Points

The best type of tank for mixing is cylindrical. Steam coils or other obstructions in a tank somewhat impede mixer action and often require added horsepower. Rectangular tanks usually require more power than plain cylindrical tanks.

Where mixers are to be used with different sized propellers, the motor H. P. used should be equal to the largest propeller required. With rectangular or special shaped tanks, it is often best to use two mixers of smaller size.

Among the precursors of the modern emulsifying machine, is found a type which forced the components through a small opening or between two surfaces in motion.<sup>8</sup> Another type forced a preliminary emulsion through capillary tubes at high pressure.<sup>4</sup> A third type continuously forced the preliminary emulsion through a conical spring valve.<sup>5</sup> The "colloid mill" invented by Plauson, forces the liquid between a rapidly moving rotor and a stator. The term "colloid" mill is a misnomer as particles of colloidal size are not necessarily produced by it.

### Colloid Mills

Colloid Mills,\* through popular acceptance and wide usage, have come to be defined as—machines whose functions are to disintegrate, disperse, emulsify and mix, otherwise immiscible materials. A true colloid mill should be capable of so reducing the particle size of the materials being treated that they will remain in suspension without separation or settling for long periods of time. In achieving stability, of

<sup>&</sup>lt;sup>8</sup> Marix, Fr. 218,946, 218,947 (1892).

<sup>&</sup>lt;sup>4</sup> Gaulin, Fr. 295,597 (1899). <sup>5</sup> Gaulin, Brit. 22,875 (1904).

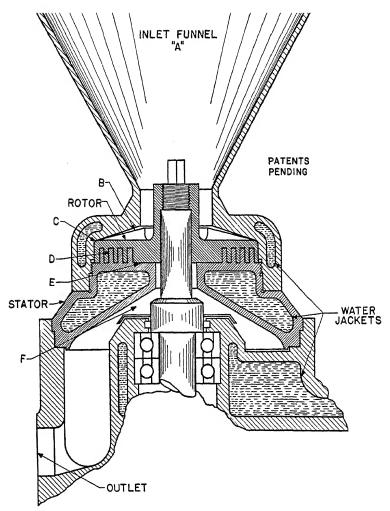
<sup>\*</sup> Courtesy of C. O. Bartlett-Snow Co., Cleveland, O.

emulsions or dispersions, it is sometimes necessary to resort to the use of various chemicals, gums, etc., but it is the responsibility of the colloid mill to produce the initial fineness of dispersion or emulsification.

Any attempt to list all of the industries in which the colloid mill does, or should, find a place would be a huge task, as new uses are found almost daily for this versatile equipment. It is a long step from asphaltic materials and road surfacings, to dairy products, mayonnaise and beverage concentrates, but the colloid mill is used in producing all of these. Automobile and furniture polishes, wax and resin paper coatings, jellies, flavorings, vitamin products, various pharmaceuticals and cosmetics, are only a few more of the many emulsions which benefit from treatment in these mills.

The fineness of emulsions or colloidal suspensions depends on the amount of "shearing" which the particles or globules in the mixture receive. Whether this is brought about by sudden violence such as is said to have produced the emulsions found in the oil fields, or to the slow grindings of thousands of centuries which produce the familiar colloidal river muds and clays, is of little moment. Also, it matters little whether the dispersions are produced by hydraulic or attritional shear providing only the amount of shear has been sufficient.

Colloid mills, in the past, have depended for their results almost entirely on "hydraulic shear." By passing materials to be treated between the faces of closely adjusted rotors, or rotors and stators revolving at high speeds, "hydraulic-shear" acts on the agglomerates, oil or fat globules, breaking them up and dispersing them. Because of practical limitations, the areas of the rotor faces have been relatively small. Many different types of rotors have been designed with a view to overcoming this limitation and creating increased rotor area—some attempts having been more successful than others.



Diagrammatic view of a Bartlett-Snow Triple Action Colloid Mill showing—"A" the inlet funnel;—"B" the impellers or vanes on top of the rotor;—"C" the close clearance between the rotor and the housing where the first processing action takes place;—"D" the interlocking rings on the bottom of the rotor and the top of the stator where the second processing action takes place;—"E" the smooth section between the stator and the rotor where the third processing action takes place;—and "F" the chamber through which the material drains to the discharge outlet.

How much of the dispersive action in a colloid mill results from the direct attrition of the rotor face, how much from inter-particle impingement, and how much from "hydraulic-shear," has remained problematical. Although some "grinding" action and reduction of particle sizes has been claimed for colloid mills, several of the foremost authorities on the subject claim that colloid mills do not grind or reduce the size of the particles, but merely disperse the agglomerates.

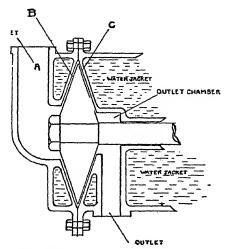
Generally speaking the essential considerations of a colloid mill—listed in order of importance—are:

(1) The efficiency or thoroughness with which the machine disintegrates, disperses, emulsifies or mixes the material, (2) the facility for feeding the machine and for removing the processed material, (3) the ease of disassembly, cleaning, and re-assembly, (4) the absence of packing, glands, etc., (5) low horsepower requirement per gallon of throughput.

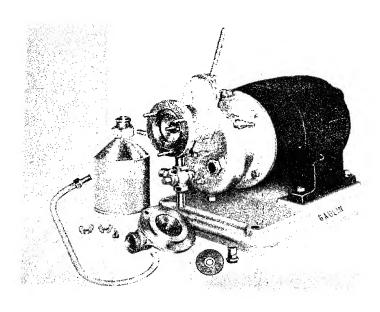
### The Manton-Gaulin Colloid Mill

The material to be treated is introduced at A and is forced through the first stage gap B, by impellers located on the front rotor face. These impellers also act as cutters and shredders, breaking down and thoroughly disintegrating the material as it passes through. In this gap the material receives a treatment given by no other mill. The pressure built up by the impellers forces the material across the outer edge of the rotor, then inward across the second stage, or back face C, where it receives another and finishing treatment.

It should be noted that in its passage through gap "C," the product being treated is travelling counter to the path of centrifugal force. This is of extreme importance. Material



The Manton-Gaulin Colloid Mill



Gaulin 2" Mill Disassembled

in the second stage enters the gap at the periphery, where it covers the maximum rotor area. From here it is crowded and pushed into the diminishing gap area by pressure, and emerges at the center of the rotor, at practically no pressure. In its entire passage through the second, or finishing stage, the material receives the maximum possible rotor action. The second stage "C" is adjustable by means of an adjusting dial which is calibrated in thousandths of an inch. All adjustments can be made while the mill is running.

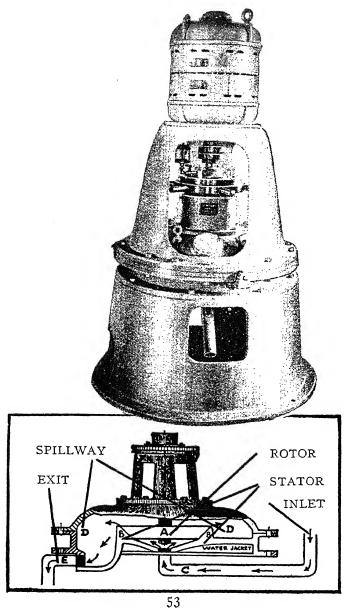
### The Premier Colloid Mill

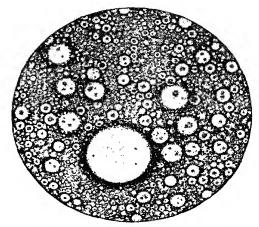
A study of the sectional drawing of the liquid type Premier Colloid Mill shows the material passing through this mill. Entering through inlet (c), passing through the clearance between the rotor (a) and the stator (b), materials are discharged through the spillway (d).

## Homogenizers

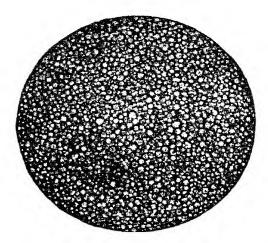
The process of homogenization consists essentially in passing the product under pressure between ground surfaces. High velocity, hydraulic shear, pressure release and impact rend the disperse phase (or material to be emulsified or dispersed) into a very fine state of sub-division. The particles or globules are reduced to one micron and less in diameter. (One micron is equivalent to approximately 1/25000 of an inch.)

In the homogenizing process, the reciprocating action of a plunger type pump forces the product between the valve seat and the valve. Pressure is applied by a handwheel which restricts the valve gap and the material is forced through by the pressure which is built up. Since the pressure is positive, receptacles on levels much higher than the homogenizer may be filled directly from the discharge pipe.

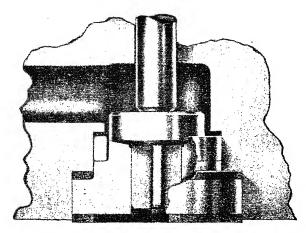




Ordinary Emulsion Magnified X 1,000



Same Emulsion Processed through the Premier Mill Magnified X 1,000



The above illustration shows the homogenizing valve for the Gaulin Laboratory Homogenizer. The valve and valve seat are ground to fit each other perfectly. The valve has a long closely fitted guide or pilot to insure accurate seating.

### CHAPTER IV

#### STABILITY

UNDER the microscope, emulsions appear to consist of small drops of oil suspended in a liquid. The droplets are surrounded by soap pellicles which not only impede their coalescence but also keep them uniformly dispersed. Dilution breaks emulsions because a certain quantity of soap becomes dissociated in the water. The elasticity and toughness of the layer protecting the droplets is of great importance to the stability of an emulsion. These properties are not the same with all emulsifiers. For example, under the same conditions, the pellicle of sodium oleate is much more resistant than that of sodium stearate. The resistance of sodium stearate is even inferior to that of oleic acid alone. When oleic acid soap is added to a colloidal stearic acid soap solution in water, it is the stearate that is dispersed. To make the most stable emulsions, an emulsifying agent is required that will give pellicles which are most resistant to the conditions to which the emulsion must be exposed. It is also necessary that the product to be emulsified, is either a liquid or becomes dissolved during the preparation of the emulsion. It must also be subdivided mechanically into very small particles, the smaller the particles the more stable the emulsion. Other factors in stability are the viscosity of the dispersion, the proportion and solubility of the emulsifier in the dispersing means, the stability of the emulsifier in the air, with heat, in the presence of acids and salts, and its ability to form elastic and very thin adhesive layers. The specific gravity of the dispersed phase and dispersion medium influences the stability of emulsions. The greater the difference in specific gravity, the greater the tendency for the emulsion to break. The greater the volume of the droplets, the less stable the emulsion. With the same proportions of oil, water, or soap the emulsion may be fluid, or more or less dense, according to the size of the droplets. The addition of a larger proportion of water makes some emulsions more stable and others less stable. The activity of emulsifiers can be increased by adding other colloids like glue, casein or starch, which render the pellicle, surrounding the emulsified substances, more resistant.

The strength and compactness of the interfacial film are the most important factors favoring stability of an emulsion.<sup>1</sup> Most of the other influences are effective only insofar as they modify the properties of the film. Among these subsidiary factors the concentration of the emulsifying agent is significant. The stability of an emulsion of moderately high oil concentration is seriously affected by the addition of electrolytes, only if the amount of agent is insufficient to form a coherent film around the oil globule. The typical stabilized emulsion of the oil-in-water type possesses hydrophilic properties, especially when there is an excess of emulsifying agent, and may be regarded as a combination of and an intermediate between a hydrophilic and a hydrophobic colloid. Although interfacial tension plays a doubtful role in the stability of emulsions, the interfacial adsorption, which accompanies it, is an important stability factor. Viscosity by itself cannot explain emulsion stability, even though viscous emulsions are generally more stable than mobile ones; high viscosity is regarded as a symptom rather than as a cause of stability in emulsion systems.

As early as 1923 Finkle 2 suggested that the relative volume of the polar group should play an important part in determining the type and stability of the emulsion. If, e.g., the metal radical in a soap occupies a large volume in the aqueous phase, the close packing will form a surface convex from the aqueous side (hence an oil-in-water emulsion). The larger the volume of the metal radical the more convex the surface will be (hence the smaller the drop and the more stable the emulsion). If the metal is multivalent, the volumes of the attached carbon chains will be relatively larger than that of the metal, and the close packing will make the surface concave towards water (hence a water-in-oil emulsion), and the more concave the surface the higher the valence and lower the volume of the metal. A series of hydrocarbon and water emulsions, with oleates and stearates of cesium, potassium, sodium, calcium, silver, magnesium, zinc, aluminum and iron, showed this type of emulsion and its stability to be consistently correlated with the atomic volumes of the metals. The drop sizes of emulsions containing cesium, potassium and sodium stearates were similarly consistent.

Schulman reports optimal stability of an emulsion of heavy refined mineral oil results if: the interfacial film is electrically charged; the interfacial film is stable and completely covered with charged molecules. Such conditions are met by one phase being oil-soluble and the other ionizable in water. The stability of interfacial films runs similarly to that of the same films at the air-water interface and depends on the Van der Wales forces of attraction between non-polar groups and on the inter-action of the respective head groups.

<sup>&</sup>lt;sup>2</sup> Finkle, J. Am. Chem. Soc. 45, 2780 (1923).

# Viscosity

Emulsions made with natural colloids (e.g., gelatin, gum tragacanth or egg yolk) are more viscous than those made with soaps. Where excessive dripping or penetration of an emulsion is undesirable, a more viscous emulsion is preferable. In any given formulation it may be assumed that the greater the viscosity, the better is the emulsification providing that all other conditions are satisfactory.

The following formula, developed by Stokes, shows the effect of viscosity on the settling rate of dispersed particles.

$$V = \frac{2r^2(s-s^1)g}{9n}$$

V Rate of settling

r Radius of dispersed particle

s Density of dispersed particle

s1 Density of liquid phase

g Gravitational constant

n Viscosity of liquid phase

Mathematical formulae for predicting emulsion viscosity have also been given by a number of other investigators.<sup>2</sup>

Increasing the viscosity of emulsions, when necessary, is accomplished by changing the ratio of the phases, increasing the amount of emulsifier, inversion of the emulsion type or the addition of small amounts of specific materials such as alcohols, cresols, eugenol, thymol, carvacol, glycerol, esters, and so forth. If too large an amount of the latter is added breaking may result.

Next to concentration, the stabilizer used seems to be of most importance in determining the viscosity of any phase pair. Stabilizers that give the best emulsions (most readily

<sup>&</sup>lt;sup>2</sup> Clayton, Theory of Emulsions, p. 130, P. Blakiston Son & Co. (1935); Patrick in Taylor's Treatise on Physical Chemistry, Vol. II, p. 1314, D. Van Nostrand Co. (1924); Monson, J. Ind. & Eng. Chem. 30, 1287 (1938).

emulsified) also give emulsions of the lowest limiting relative viscosity.<sup>16</sup>

The viscosity of gelatin emulsions is reduced by hydrolysis on aging or heating and also by exposure to ultrasonic waves. 16

Irregular viscosities of the same emulsions are most pronounced when freshly made. Exclusion of air from the emulsion gives similar viscosity.<sup>1d</sup>

Viscosity of emulsions of the same pair of phases (paraffin oil and water with sodium oleate) is not dependent on particle size or size distribution within wide limits. Viscosities of emulsions of the same continuous phase and different disperse phases are almost independent of the disperse phase. The viscosity of emulsions decreases with increasing velocity gradient and is independent of it beyond a certain limit. 16

## Viscosity of Colloidal Solutions in Presence of Electrolytes

The available data relative to the influence of electrolytes on the viscosity of colloidal solutions appear to show that the viscosity of the solution increases with the thickness of the layer of water covering the colloidal particle, an oil emulsion representing a limiting case. The addition of an electrolyte, in small quantities, increases the potential of the electric double layer at the surface of the particle, whereas, in excess amounts, it diminishes the charge by coagulation. Since the former effect increases the viscosity and latter diminishes it, a minimum on the viscosity-electrolyte concentration curve is anticipated.

In soap and alkali salt solutions the lowering of viscosity by small additions of electrolytes, is due to loss of water by

<sup>&</sup>lt;sup>1h</sup> Broughton, J. Phys. Chem. 42, 253 (1938).

<sup>&</sup>lt;sup>1c</sup> Porter, J. Ind. & Eng. Chem. News Ed. 12, 748 (1940). <sup>1d</sup> Narayanaswamy, J. Ind. Inst. Sci. 17A, 75 (1934).

<sup>&</sup>lt;sup>1e</sup> Sibree, Trans. Faraday. Soc. 27, 161 (1931).

the colloidal particle, and consequent shrinkage. This loss of waters occurs through osmosis.

On the other hand the lowering of the viscosity may not be due to shrinkage, but to loss of water caused by the increase of charge resulting from the preferential absorption of either positive or negative ions from the solution.

At higher concentrations, coagulation occurs, accompanied by water absorption, and the viscosity increases. If no absorption of water occurs, the viscosity does not increase with coagulation, therefore the viscosity is closely related to the homogeneity of the coagulum.

Colloidal proteins have high viscosities, and contain solid particles that occlude large volumes of water, which can no longer function as solvent. It is very difficult to find a theory of inorganic colloids which covers the facts, but it is suggested that the addition of an electrolyte may cause an increase of cataphoretic potential, with corresponding diminution in viscosity. The viscosity of natural W/O petroleum emulsions is increased by ferric ions. Is

### Phase Volume Ratios

The relative volumes of the phases effect the type and stability of an emulsion; e.g., if in making margarine, the milk is run into the large volume of oil, while stirring, a W/O type results.<sup>1a</sup> When the oil is mixed into the milk a O/W type forms.

In emulsions made with soap there is no particular limit to the amount of soap, but the amount of water must vary within a fairly close range to produce a proper hydrating effect and properly orient in conjunction with the soap and

<sup>1</sup>f K. C., Sen Kolloid-Z 38, 253-7 (1926).

<sup>&</sup>lt;sup>1g</sup> Sherrick, J. Ind. & Eng. Chem. 12, 137 (1920).

<sup>&</sup>lt;sup>18</sup> Clayton, Margarine J. 2, 13,503 (1920).

<sup>&</sup>lt;sup>1h</sup> Brown, Textile Colorist, 57, 516 (1935).

The shape of the mixing vessel affects the critical amounts of the phases. 1c

## Creaming

Creaming is the separation of a concentrated layer of the disperse phase.

A simple test for creaming, in an opaque emulsion, is to dip a finger in it. If there has been creaming, some of the emulsion will stick to the finger instead of draining off quickly.<sup>2a</sup>

Mathematically it is computed that this cream layer contains 74% by volume of the disperse phase. Creaming is dependent on the relative specific gravities of the oil and water phases. In O/W emulsions, creaming takes place at the top if the oil is lighter than water. If the oil is heavier, the cream forms at the bottom.

The speed of cream formation depends on the specific gravity of the oil, viscosities of both phases, globule size, quantity and solubility of emulsion and a number of other variables.

The velocity, with which a particle of dispersed oil rises or creams, is directly proportional to the difference in density between the oil and water phases and the square of the diameter of the oil particle. It is inversely proportional to the viscosity of the aqueous phase. Thus, if the diameter of the oil particle is reduced one half, a given amount of creaming would require four times the time interval. Emulsions of sufficiently small particle size may never cream, e.g., homogenized milk.

Retardation of creaming is helped by agitation or by the addition of a heavy or light material (as the case may be) to the oil phase. Preferably this addition should be insoluble

 <sup>&</sup>lt;sup>1c</sup> Crockett, J. Ind. & Eng. Chem. 9, 969 (1917).
 <sup>2a</sup> Brown, Textile Colorist, 57, 515 (1935).

in the water phase. Thus, in a mineral oil, soap, water emulsion the densities of the phases are equalized by the addition of cresol and creaming is retarded. Similarly, carbon tetrachloride is used to prevent creaming. In W/O emulsions creaming is in the opposite direction to that previously mentioned.<sup>2b</sup>

## Hydrophilic Emulsions

Where hydrophilic colloids are used the emulsions have a larger particle size than when soap is used, but are more stable.<sup>3</sup> The former are less affected by addition of an electrolyte and rise of temperature. Their emulsions are more viscous and may even gel. Decreasing this viscosity tends to decrease stability.

The efficiency of an emulsifying agent is due to its ability to form a strong solvated membrane at the oil-water interface. Thus, agents which gel readily form strong membranes, giving stable emulsions.

Where gums are used, high viscosity emulsions are formed. This indicates that much of the gum does not remain at the interface, but is in the dispersed phase. Although this produces a coarse emulsion, it favors stability because creaming and agglomeration of oil particles caused by kinetic impact are greatly reduced.

Hydrophilic emulsifiers, that do not form gels, form a fine particle size emulsion, but because of the low viscosity of the continuous phase, relative instability results. This low viscosity is due to the fact that no solvated membranes are formed, and there is less tendency to form an oriented adsorbed layer than when soap is used.

Deterioration of hydrophilic stabilized emulsions, which

<sup>&</sup>lt;sup>2b</sup> Modern Cosmeticology. Harry p. 7. Chemical Publishing Co., Inc., Brooklyn, N. Y.

<sup>&</sup>lt;sup>3</sup> J. Soc. of Chem. Ind. 59, No. 9, 185.

appear stable when made, is often caused by the gradual growth of the droplets of the dispersed phase, without actual separation. Lecithin, however, acts more like soap in this respect, i.e., showing an oil separation on aging.

Tragacanth stabilized emulsions of vegetable and mineral oils are especially stable between pH 1.9 and 2.3. Acacia has stability range between pH 2–10 and shows a higher dispersion.

A stable emulsion is one in which there is no separation or concentration of phases as evidenced by formation of oil droplets coalescing to form an oil or water layer, and no creaming due to concentration of the dispersed phase.

Homogenized soap emulsions of oils deteriorate not by gradual globule size increase, but by distinct oil separation.<sup>4</sup> All globules larger than 7.5u are essentially unstable.<sup>1</sup> Similar emulsions, made by shaking, are coarser with the initial size of the globules being greater than 7.5u. These globules grow by coalescing without separating free oil.

The particle size of emulsions varies within wide limits. In transparent emulsions the particles are smaller than the wave length of light (less than  $10^{-5}$  cm.). Bluish milky emulsions are composed of particles of the size of  $10^{-4}$  to  $10^{-5}$  cm. White emulsions contain particles  $10^{-4}$  cm. and larger.

Thus, the transparency or color of an emulsion indicates the particle size and in some cases is an indication of stability and certain physical characteristics.

The stability of an emulsion can be predicted if the size distribution of the globules is known. The method of determining distribution (size-frequency) is as follows: the emulsion is diluted with an equal amount of strong gelatin solution. This is examined at about 1100 diameters through the

<sup>4</sup> Billings, Amer. Dyestuff Rept. p. 267 (May 1938).

<sup>&</sup>lt;sup>5</sup> Technical Aspects of Emulsions, p. 52, 53, Chemical Publishing Co., Inc., Brooklyn, N. Y.

microscope. The approximate size of the globules is determined with a squared eye-piece, calibrated by means of a stage micrometer. Counts of various size groups are made and compared with counts of similar emulsion samples. To keep errors at a minimum, at least 1000 globules are measured and counted, using at least two slides and several noncontiguous fields on each slide. It is also necessary to vary the focus so as to examine different depths of the film.

In storing emulsions care should be taken to prevent evaporation which will cause deterioration. Change in composition due to chemical interaction between the ingredients or with the container may also cause break-down.

In the preparation of emulsions by use of a hand homogenizer, a mixture of acacia and tragacanth (3:1) gives better results than either gum alone. The mixture of gums gives emulsions that are whiter than those made with tragacanth alone, and shows less creaming than those made with acacia alone. These results hold true for cod liver oil, linseed oil and mineral oil but not for castor oil. The use of tragacanth in combination with acacia is of no advantage if the emulsions are made with a mortar and pestle. From the results of viscosity tests, it appears that the beneficial results obtained by addition of tragacanth in certain cases, are due to factors other than viscosity alone.

When using gelatin, pH is also a very important consideration. The electrical charge, as well as the protective membrane of gelatin formed on the droplets, determines the stability. At low emulsifier concentrations the electrical charge becomes an important factor. Changes in stability of gelatin-stabilized emulsions at various pH values are related to changes in surface tension and viscosity of gelatin sols with changing pH.<sup>8</sup>

<sup>&</sup>lt;sup>7</sup> Technical Aspects of Emulsions, p. 112.

<sup>8</sup> Limburg, Rec. Nav. chim. 45, 772,854,875 (1926).

A benzene emulsion, stabilized with gelatin at its isoelectric point (when surface tension and viscosity is lowest), showed the greatest stability. Correlated data of does not indicate that in emulsions of this type the electrical charge is an important factor. Gelatin emulsions will vary because of variations in the gelatin and because many are not made at a uniform pH.

Saponin stabilized emulsions are very sensitive to calcium, whereas gelatin stabilized emulsions are not broken by calcium chloride. Agar and lecithin stabilized emulsions are coarsened and show lower stability when treated with calcium chloride. It appears that emulsions made with hydrophilic colloids which give the greatest viscosity are more stable to calcium ions. The addition of small amounts of acid has a similar effect.<sup>10</sup>

Thus, emulsion films can be stabilized and toughened by these and other various additions. So, the addition of a small amount of sodium sulfate to a kerosene emulsion with gelatin increases stability.<sup>11</sup> Sodium silicate is likewise helpful in certain soap emulsions.

Octane emulsions with certain soaps are less stable when freshly made.

Benzene emulsions made with soap are adversely affected by addition of oleic acid to the oil phase or alkali or salt to the aqueous phase.<sup>12</sup>

Inversion and then instability results when univalent cations equal to those of the soaps are introduced.<sup>13</sup>

For benzene, toluene and xylene, potassium oleate is a better emulsifier than sodium oleate. Sodium chloride breaks these emulsions more readily than potassium chloride. Sta-

<sup>&</sup>lt;sup>9</sup> J. Amer. Chem. Soc. 53, 2898 (1931).

J. Russ, Phys. Chem. Soc. 59, 1061 (1927).
 Introductory Colloid Chemistry. Holmes p. 75 (1934).

<sup>&</sup>lt;sup>12</sup> Harkins, Fifth Colloid Symposium Monograph p. 19 (1928). <sup>18</sup> Tartar, J. Phys. Chem. 33. 435 (1929).

bility of xylene emulsions with soap is improved by phenol or its derivatives.<sup>14</sup>

The addition of certain amounts of chloroform stabilizes electro-negative colloids but not those that are electro-positive.<sup>15</sup>

Glycerin stabilizes oil emulsions with soap and Irish moss. 16 Gum acacia stabilizes oil emulsions with soap.

Water-oil emulsions may be stabilized with a dammar solution.<sup>17</sup>

Stability of olive, castor, and cod liver oils is increased by a little free fatty acid, using caustic soda as the emulsifier. W/O emulsions of these oils with calcium and barium hydroxide are coagulated by excess alkali. Emulsification is favored by the addition of gelatin or gum arabic.

Emulsions containing free alkalies, that may be stable when made or kept in air-tight containers, develop a scum on the surface due to absorption of carbon dioxide and begin to break at the surface. A critical temperature exists below which no scum forms.<sup>19</sup>

Precipitates in emulsions may be due to excess of a sparingly soluble ingredient or the formation of an insoluble reaction product, e.g., a lime soap formed from dissolved calcium compounds and soap. Such reaction products may form slowly, but will definitely effect stability.

In W/O emulsions, containing coal particles, the latter are held in suspension at the water oil interface. The coal particles tend to displace the emulsifier and break the emulsion.<sup>20</sup>

Stability may be increased by proper adjustment of ion-

<sup>&</sup>lt;sup>14</sup> Viekhhertz, Colloid J. (U.S.S.R.) 2, 133.

<sup>15</sup> Sabbatini, Atti. 1st Veneto 87, 321 (1927).

<sup>&</sup>lt;sup>16</sup> Crockett, J. Ind. & Eng. Chem. 9, 969 (1917).

<sup>&</sup>lt;sup>17</sup> Holmes, U. S. 1,429,430 (1922).

Iselin, Boll Chim. farm. 67, 225 (1928).
 Davey, Phys. Rev. 11, 138 (1918).

<sup>&</sup>lt;sup>20</sup> Hedrick, Ind. Eng. Chem. 33, 1057 (1941).

adsorption. Hydrogen ion adsorption in phenol water emulsions is an example.<sup>21</sup>

Very acid sulfonated vegetable oil gives stable mineral oil emulsions of pH 5.8–9.0. Sulfonated cod oil gives stable emulsions only at pH 7.8 to 12.0. These emulsions dilute readily and are broken by more than 3% salt.<sup>22</sup>

A mixture of alizarin oil and spindle oil gives an emulsion, the stability of which increases (pH 6.9–7.0) with the increase of alizarin oil (from 10 to 40%). A mixture of sulfonated train oil and spindle oil, in general, gives more stable emulsions than a similar mixture with alizarin oil. The stability of a 5% emulsion of a mixture containing 50% spindle oil, 40% sulfonated train oil and 10% sulfonated petroleum increases with the increase in the pH. The stability of these emulsions is not affected by temperatures within 20–40°.23

Vegetable and mineral oil emulsions prepared with gum acacia are stable between pH 2–10. Changes of pH do not affect the relative viscosities or surface tensions of these emulsions. Alkalies are detrimental. Such emulsions made with gum tragacanth are most stable at pH 1.9–2.3 and are unstable when alkaline. They also change in particle size and viscosity with change in pH. Stability decreases with increase in particle size.<sup>24</sup>

For some purposes a quick breaking emulsion is desirable, as in agricultural sprays. Here the emulsion should break as soon as possible after application to the plant, and thus release the active ingredient. Such quick breaking emulsions are usually unstable and are made up just before use. Quick breaking emulsions usually contain an insufficient amount of emulsifying agent.<sup>25</sup>

<sup>&</sup>lt;sup>21</sup> Ellis, Z. phys. Chem. 80, 597 (1912).

<sup>Woodroffe, J. Inter. Soc. Leath. Trades Chem. 12, 419 (1928).
Pehelin Tz. Nauch. Sbornik Rabot 4, 111 (1934).</sup> 

 <sup>&</sup>lt;sup>24</sup> Krantz, J. Amer. Pharm. Assoc. 15, 83, (1926).
 <sup>25</sup> Technical Aspects of Emulsions, p. 73.

An emulsion may be stable in W/O type and may become unstable due to inversion caused by shaking in transit. Instability of soap emulsions by inversion of type because of dilution with hard water and formation of lime soaps,<sup>26, 27</sup> is also common.

The stability of mayonnaise is increased by the incorporation of finely divided mustard powder.<sup>28</sup>

The thickness of the emulsifier interface is one of the criteria of stability. The initial concentration of emulsifier at the interface is not the minimal amount necessary; the important factor being the concentration after adsorption has taken place.<sup>29</sup> An emulsion of globules protected by an expanded film is unstable. A compact, oriented, monomolecular film gives greater stability.<sup>30</sup> The greater the resistance of this layer to rupture, the greater the stability of the emulsion.<sup>31</sup> Soap films tend to be monomolecular, either attenuated or compact.

If the emulsifying agent is peptized too much, by the continuous phase, it will not form a protective film at the interface and an emulsion may not form. This is the case when hydrous ferric oxide is used to make a benzene-water emulsion. If a little dilute sodium chloride solution is added, a crude emulsion is formed.<sup>32</sup> A slight increase in salt produces greater stability.

A certain type of mechanical treatment may break as well as make an emulsion. Thus, if the droplets formed travel in parallel lines at different velocities along too great a distance, orthokinetic coagulation takes place.

<sup>&</sup>lt;sup>26</sup> Ibid., p. 94.

<sup>&</sup>lt;sup>27</sup> Ibid., p. 95.

<sup>&</sup>lt;sup>28</sup> Ibid., p. 96.

<sup>&</sup>lt;sup>29</sup> Fischer, J. Phy. Chem. 36, 98 (1932).

<sup>&</sup>lt;sup>30</sup> Markins, J. Amer. Chem. Soc. 51, 1674 (1929).

<sup>&</sup>lt;sup>31</sup> Technical Aspects of Emulsions, p. 112. <sup>32</sup> Briggs, Ind. & Eng. Chem. 13, 1008 (1921).

The stability of a delicate emulsion in a closed container is affected by the enclosed air in two ways; by mechanical means, and by changes in concentration through vaporization and condensation due to temperature changes.<sup>33</sup>

Many emulsions are broken by freezing. Freezing breaks them by destroying the membrane of emulsifier surrounding each globule, rather than by the mere freezing of the continuous phase. <sup>33a</sup>

Some emulsions can be frozen solid and thawed without breaking. Supercooling without actual freezing is often possible, but freezing usually results in breaking. On freezing and thawing, with resultant breakdown, the emulsifier may disappear from the water phase.<sup>38b</sup>

Heat may produce profound chemical or physical changes which may break an emulsion. Heating of emulsions made with hydrophilic agents usually increases globule size. Agar stabilized emulsions break at temperatures above the gelling point of agar dispersions. Lecithin emulsions are, however, less affected by heat.

For this reason, heat is only used to render a very viscous or solid phase fluid. In the latter case, the water phase should be heated to about the same temperature, to avoid chilling and solidifying the solid phase. No higher temperatures should be used than are necessary.

Aging decreases specific interfacial area. This decrease is greater with natural colloids than with soap. Initial size frequency analysis of the former is not necessarily indicative of stability, i.e., a finely dispersed emulsion of the former is naturally less stable than a coarsely dispersed emulsion.

Time shows the effects of very slow chemical reactions as changes of state. Similarly sedimentation or separation,

<sup>33</sup> Nagelstein, Kolloid-Z-83, 224 (1938).

<sup>&</sup>lt;sup>88a</sup> Rochow, Ind. & Eng. Chem. 28, 1926 (1936). <sup>88b</sup> Technical Aspects of Emulsions, p. 146 (1936).

because of the difference in density, may become evident on aging.

Oil emulsions with Irish moss are broken by a trace of soap. Addition of a little glycerol prevents breaking. Larger amounts of soap aid emulsification. Better emulsions result when all the water is added at once to the mixed oil and Irish

# Causes of Instability

Many persons are completely at sea when a preparation separates. The causes of separation are not so numerous. Some of them, of course, are due to physical reactions about which little is known. Among the causes encountered are:

DECOMPOSITION: This usually occurs when mucilages, albumen and animal products are used as emulsifying agents, and is due to the lack or an insufficiency of preservative.

INCOMPATIBILITY: This occurs when the emulsion contains an ingredient which is incompatible with the emulsifying agent and causes it to precipitate, e.g., borax in an emulsion containing gum acacia; mineral acids in emulsions containing albumen. Another very common instance of incompatibility occurs in connection with an attempt to make an acid emulsion with a soap emulsifying agent.

Insufficient Dispersion: This is due to failure to reduce the oil globules to a very minute size. This is a deficiency caused by lack of adequate equipment or proper technique. There are any number of emulsions that will separate on standing when mixed in the ordinary way but will stand up indefinitely when mixed properly as, for instance, in a colloid mill or homogenizer.

EXCESSIVE DISPERSION: This is the reverse of the foregoing and is due to such fine dispersion that the properties of

<sup>84</sup> Crockett, J. Ind. Eng. Chem. 9, 967 (1917).

the emulsifying agent are destroyed. This condition frequently occurs in connection with preparations containing vegetable mucilages, which should be mixed at low speeds with a paddle type mixer.

ELECTROLYTES: The presence of salts especially those of calcium, magnesium, aluminum, copper, iron, zinc, bismuth, mercury, are likely to destroy emulsions, but in some instances very small quantities can be added after the emulsion has first been formed.

TEMPERATURE: Numerous emulsions will break if exposed to low temperatures; others will break if exposed to high temperatures. Emulsions containing soap are likely to be sensitive to cold; those containing animal products are likely to be sensitive to heat. Sometimes, a temperature variation of twenty degrees one way or another will cause separation, as for instance, when emulsions are heated by attrition in going through a colloid mill, or by pressure in a homogenizer.

INSUFFICIENT VISCOSITY: Many emulsions will separate if not sufficiently viscous. This can be remedied by the addition of a thickening agent that will raise the degree of viscosity.

IMPROPER PROCEDURE: Some emulsions must be mixed slowly; others must be mixed rapidly. Some will require the making of a primary emulsion first which can be diluted after emulsification has been effected. Chemicals like phosphorus, sulphur, camphor, creosote, cannot be added to the batch directly but if first dissolved in a fixed oil, can be emulsified very readily.

FAILURE TO DISTINGUISH TYPES: Attempts to add water to water-in-oil type emulsions or oil to oil-in-water types will cause separation. Many fail to distinguish between these two emulsion types. They assume that because the emulsion contains more oil than water they can add more oil to it, whereas

an emulsion can consist of ninety per cent oil and ten per cent water and still be an oil-in-water emulsion.

IMPROPER EMULSIFYING AGENT: No one emulsifying agent is suitable for all emulsions. The user should know what emulsifying agents are best for his particular emulsion. Often a stable emulsion can be secured by changing its type, that is from oil-in-water to a water-in-oil, an effect which can be accomplished by changing the emulsifying agent.

## Summary

## Emulsion instability may be caused by:

Improper ratio of oil and water phases.
Incorrect amount of emulsifier.
Impurities in oil or water phases or emulsifier.
Reaction between two or more components and/or container.
Overheating
Excessive evaporation
Freezing
Mechanical shock or vibration
Improper balancing of densities
Time of mixing
Absorption of gases or foreign particles.

## Testing Stability of Emulsions

- a. The quickest way of testing stability is to use a small laboratory centrifuge. Put the samples in tubes and keep a record of the time required to break them as against a known standard. In order to see how emulsions will resist cold, put samples into a refrigerator and keep the temperature around the freezing point for a couple of days. To check heat resistance put samples into a thermostatically controlled electric oven and keep them there for a couple of days at a constant temperature of 110°F.
  - b. A glass cylinder 25 millimeters in diameter with a

bulbous extension at the bottom is fitted with a side cock and rests in a wooden support. The cylinder is charged with 100 cubic centimeters of the emulsion and placed in a drying oven to settle at a definite temperature and for a definite time. After 20 cubic centimeters of the liquid has been removed and discarded, 10 cubic centimeters is withdrawn into a tared aluminum beaker and weighed before and after evaporation into a drying oven, giving the moisture content. If the emulsion contains B per cent of dispersed, and a is the percentage of the dry substances of the phase, and b the weight of the evaporated moisture, then the content of non-fatty part in the emulsion is:

$$b(100+a)/a$$

and the stability of the emulsion is: 86

$$b(100+a)/B$$

c. Two copper plates are kept apart by 2 pieces of glass of about 0.2 millimeter thickness with a rubber band around the whole. The breaking of an emulsion is indicated by the glowing of an electric lamp of 50 candle power with alternating current of 120 volts and 50 cycles passing through the electrodes and the emulsion at 40–50°.

The efficiency of various methods of preparation of emulsions and emulsifying agents is shown by different stabilities of the emulsions.<sup>27</sup>

A finer W/O emulsion of vegetable oil with milk, salt, (margarine) is made by supercooling before adding the emulsifier.<sup>38</sup>

Attempts to homogenize an emulsion containing more than 74% of oil (the disperse phase) resulted in the production of

<sup>&</sup>lt;sup>36</sup> Bochko, Maslo. Zhiro. Delo. 11, 489 (1935).

<sup>&</sup>lt;sup>37</sup> L. Pick, Chem. Obzor *3*, 202-3 (1928). <sup>38</sup> Newton, U. S. 2,232,401 (1941).

a new emulsion containing approximately the volume of oil required for the globules to be in the closest packing when of uniform diameter. Microscopic examination of the new emulsion produced by homogenization showed that the globules were small and of almost uniform diameter. It was very difficult to increase the oil content of such an emulsion even with additions of 1 cc. of oil and shaking after each addition. Emulsions containing less than 72% of benzene or 70% of toluene (the two compounds used in the experiments) did not show any tendency to decompose on being passed through the homogenizer, but instead in every instance, an emulsion of greater stability was produced.<sup>59</sup>

The stability of water-gas tar emulsions, in which water is the dispersed phase, is increased as the completeness of oil cracking is decreased and is affected both by the condensing and cooling methods and the quality of the tar itself. Usually the tar which condenses from the gas, in the early stage of cooling, forms the most stable emulsions. The free carbon of the tar is the emulsifying agent causing the production of emulsions, its effect however, not being the same for each of the oil constituents of the tar. In ordinary operation the most stable emulsions are formed of those tars which contain the largest percentage of saturated oils. The optimal per cent of carbon for the formation of stable emulsions varies with the oil and the tar; if present beyond this amount, there is a distinct tendency for the agglomeration and separation of the water with the formation of tar-carbon amalgams suitable for fuel. The amount of carbon to be added depends primarily upon its fineness of division. The rate of separation of water by settling or otherwise is not the same in different emulsions, but is dependent on the temperature of the emulsion, specific gravity and composition of the tar, soluble con-

<sup>89</sup> Parke, J. Chem. Soc. 1458 (1933).

stituents in the water, size of the dispersed water droplets, and other variables.<sup>40</sup>

Preparation of carron oil revealed the following: (1) The oils which yielded the most stable emulsions were olive, sesame, rape-seed and apricot kernel. Corn, cottonseed and linseed oils gave inferior emulsions.<sup>41</sup>

Emulsion breakdown in oil-in-water type creams is often caused by the use of insufficient emulsifying agent, by the manner in which the constituents are incorporated, by the use of an oil that is too light and by the reaction between the emulsifying agent and other constituents in the preparation. In the case of liquid beeswax-borax emulsions, more stable products may be secured by the addition of Castile soaps or a gum mucilage, such as quince-seed, or such emulsifying agents as glycol stearate or oleate, and so forth. Breakdown in water-in-oil types is caused by heat, chemicals, incompatibilities, lack of homogeneity, insufficient emulsifying agent and improper selection of waxes as hardening agents.<sup>42</sup>

The presence of a gas is an important factor in the stability of some emulsions. When certain liquids are freed from gas as completely as possible, and emulsification is attempted in a vacuum, no emulsion is produced, whereas introduction of a gas will yield a good emulsion. Air, as well as inert gases, may serve in this stead. It may be that thin layers of gas envelop the droplets and retard or prevent coalescence, e.g., emulsions of paraffin oil and toluene containing a gas are formed more easily and are more stable than if no gas is present.<sup>43</sup>

A difference of potential exists between the dispersed and the continuous phases. A difference of about 0.06 volts affords a fairly stable emulsion. When it becomes zero, the

<sup>40</sup> Odell, Bur. Mines, Repts. Invest. 2331, 5 (1922).

 <sup>&</sup>lt;sup>41</sup> Langenhan, Prac. Druggist. 42, 25 (1924).
 <sup>42</sup> Deakers, Drug Cosmetic Ind. 43, 36 (1938).
 <sup>43</sup> Rogowski, A. Pysik, Chem. A 166,428 (1933).

emulsion breaks—a point called the isoelectric point. The critical potential is the point, before the isoelectric point, at which the breaking of the emulsion starts. Electrolytes sometimes aid greatly in stabilizing certain emulsions.<sup>44</sup>

## Breaking Emulsions

In certain cases, it is necessary to break emulsions, e.g., (the production of butter from milk by churning). While some can be broken easily by heat, cold, electrolytes, and mechanical means, others are very resistant to breaking and must be handled in diverse ways. Cost is also an important factor which must be taken into consideration.

To decrease the stability or break an emulsion a substance should be added which breaks the film of the emulsifying agent or tends to reverse the emulsion type, e.g., calcium chloride, which inverts an emulsion made with egg yolk and breaks those made with saponin.<sup>45</sup>

As an example of chemical breaking we can take an emulsion of benzene in water, stabilized by gelatin. It is de-emulsified by a sodium hydroxide solution. If the sodium hydroxide is added immediately after emulsification the separation of benzene commences at once, whereas if the emulsion is left undisturbed for some time before the sodium hydroxide is added, the separation is inhibited for a certain period. This period of inhibition is shown to be a parabolic function of the age of the emulsion. The limiting value found for this period varies directly with the concentration of gelatin used as stabilizer over the range noted from 5 minutes in case of 0.1% to 75 minutes for 0.4% gelatin. In all cases the system was 50% by volume of benzene, and the de-emulsifier used was 0.5 normal sodium hydroxide. It is suggested, by way of explanation, that after emulsifying, the gelatin slowly forms

<sup>44</sup> Steik, Amer. Dyestuff Reptr. 25, 429 (1936).

<sup>45</sup> Berkman Egloff, Emulsions and Foams, p. 4 (1941).

protecting layers until equilibrium between the gelatin sol and the adsorbed gelatin is reached. 46

O/W emulsions are broken by many mechanical means. Milk is frothed by introducing inert gas and the cream is separated by a sieve.<sup>47</sup> Butter is separated by boiling milk at a low temperature in vacuo.<sup>48</sup>

Cream is churned in thin layers exposed to vibrational effects. 49 Wool fat is recovered from emulsions by foaming. 50, 51 Emulsions stabilized by proteins can be broken by shaking, heat or chemical denaturants. 52

W/O emulsions can be broken by such mechanical means as gravity separation; <sup>53</sup> circulating against baffles <sup>54</sup> or oscillating plates, <sup>55</sup> heating <sup>56</sup> and heating under pressure, <sup>57</sup> or by using solids, preferably wetted. <sup>58</sup> Tar emulsions are broken with finely divided inorganic powders; <sup>59</sup> petroleum emulsions are forced through infusorial earth wetted with oil, <sup>60</sup> through sand, silica, alumina <sup>61</sup> or porous diaphragms. <sup>62</sup>

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46 Nugent, Trans. Faraday Soc. Oct. 27, 1921.
  47 Ger. 314,090 (1918).
  <sup>48</sup> Brit. 333,233 (1929).
  <sup>49</sup> Perks, Brit. 322,416 (1929); U. S. 1,791,000 (1931).

<sup>50</sup> Broadbridge, U. S. 1,505,944 (1924); Karpinsky, Brit. 177,498
(1922); Bailey, U. S. 1,770,476 (1930).
  <sup>51</sup> Brit. 328,606 (1928); U. S. 1,853,871 (1932); Ger. 546,231 (1929).
  52 U. S. 1,728,011 (1930).
  53 Barber, U. S. 1,945,766 (1934); Edwards, U. S. 1,968,131 (1934);
Mahone, U. S. 1,940,726 (1933); Pink, U. S. 1,698,002, 1,698,067 (1929).
  54 Fisher, U. S. 1,942,794 (1933).

    Palmer, U. S. 1,781,076 (1930).
    Novotney, U. S. 1,968,614 (1934); Turner, U. S. 1,948,481 (1934);

Brady, U. S. 1,754,079 (1930); Moseicki, U. S. 1,710,374 (1929).
  <sup>57</sup> Champion, U. S. 1,943,367-8 (1934); Dubbs, U. S. 1,123,502 (1915);
Elliott, U. S. 1,928,282 (1933).
  58 Brit. 26,228 (1908).
  <sup>59</sup> Robinson, Brit. 268,547 (1926); Roth, U. S. 1,665,189 (1928).
  60 Zoul, U. S. 1,501,877 (1924).
  61 Van Loenen, U. S. 1,944,479-80 (1934); 1,967,601 (1934); Garrison.
U. S. 1,947,709 (1934); Eddy, U. S. 1,807,833 (1931); Pollock, U. S.
1,847,413 (1932).
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62 Trumble, U. S. 1,304,124 (1919); Haseman, U. S. 1,914,665 (1933).

In the laboratory one of the following methods usually succeeds in breaking emulsions: (1) addition of excess of the solvent in the dispersed phase, (2) addition of a third solvent miscible with each of the other two, (3) destruction of the emulsifying agent, (4) salting out, (5) filtration, (6) heating, freezing or electrolyzing, (7) centrifuging.

If the emulsion is present only as a broad layer at the interface between the two phases, it is best to draw off the emulsion separately and break it.

Examples: (1) If the emulsion is one between ether and water, take a small sample in a test tube and add successive portions of water; if unsuccessful, take a second sample and add ether. (2) A small addition of alcohol breaks an emulsion between ether and water. (3) An emulsion stabilized by soap may be broken by adding acid. (4) Formaldehyde breaks emulsions stabilized by proteins. (5) Anhydrous sodium sulphate, ammonium sulphate, or calcium chloride often salt out emulsifying agents. (6) Filtration breaks emulsions stabilized by flocculent precipitates. (5)

Underground water often forms troublesome emulsions in petroleum fields. The stability of these emulsions varies considerably.<sup>64</sup> The amount of water in these emulsions ranges from a trace to more than 60% (average 25%).

The processes for breaking such emulsions include filtration, evaporation, high voltage alternating currents, direct currents, 65 centrifuging and chemical treatment. 66

<sup>68</sup> Walter Weinberger, Am. J. Pharmacy 102, 594-5 (1930).

<sup>64</sup> Sherrick, Jol. Ind. & Eng. Chem. 12, 133 (1920).

 <sup>&</sup>lt;sup>65</sup> Cottrell, U. S. 987,114 (1911).
 <sup>66</sup> Sherrick, Jol. Chem. Soc. 67, 63 (1895).

#### CHAPTER V

## GENERAL TECHNICAL EMULSIONS

## Chemical Activity

When emulsified, water insoluble products are subdivided into very small particles which present a tremendous oil-water interface. This greatly enlarged area of contact with a reactant may greatly speed up the reaction.

When drying oils are emulsified they oxidize and dry more rapidly. Emulsions of some oils are hydrogenated by cultures of hydrogen producing bacteria.<sup>1</sup> Petroleum cracking is furthered by suitable heating of an emulsion of petroleum.<sup>2</sup> Synthetic rubbers are made by polymerization of dienes in emulsion.<sup>3</sup> Vegetable oils, in emulsion, are vulcanized by sulfur.<sup>4</sup> Pigments for latex compounding are protected by wax emulsifiable coatings.<sup>5</sup> Emulsions of heavy liquid hydrocarbons are used in making gas.<sup>6</sup> Liquid fuels are made from emulsions of petroleum oils.<sup>7</sup> Shock absorbing fluids may consist of an emulsion of lard or mineral oil and soap.

<sup>&</sup>lt;sup>1</sup> Van der Heide, Ger. 482,919 (1926).

<sup>&</sup>lt;sup>2</sup> Dubbs & Morrell, U. S. 1,744,109 (1930); U. S. 1,970,996 (1934).

<sup>&</sup>lt;sup>3</sup> Ind. Eng. Chem., 25, 1338 (1933). <sup>4</sup> I.C.I. Ltd., Brit. 313,252 (1928).

<sup>&</sup>lt;sup>5</sup> Crawford & Schoenfeld, U. S. 1,191,775 (1933).

<sup>&</sup>lt;sup>6</sup> Gas Fuel Corp., Fr. 744,602 (1933).

<sup>&</sup>lt;sup>7</sup> Vance, U. S. 1,926,071 (1933).

Many syntheses require contact between an aqueous and a water insoluble phase. The speed and completeness of a reaction are dependent on intimate contact of the reactors. Amines are made by emulsifying alkyl halides with alcohol, water and soap and allowing this to react with ammonia.<sup>9</sup> Alcohols and glycols are made by emulsifying alkyl chlorides with aqueous sodium oleate.<sup>10</sup> Ethylene dichloride is readily hydrolyzed if it is emulsified in water with ammonium linoleate.<sup>11</sup>

Acrylonitrile is polymerized while emulsified with dilute oleate.<sup>12</sup>

2, 4-dibromo, 2-methyl-butane is hydrolyzed to methyl isopropyl ketone in 80% yield by refluxing with water while stirring, and adding Twitchell's reagent or Gardinol until the solution is clear.<sup>12a</sup>

Nitration of p-cymene is accomplished by using an emulsion made with "Aquasol" or "Sulfatate." 13

The effect of emulsification on the rate of hydrolysis of amyl acetate, in alkaline solution, was determined by using several emulsifying agents. The rate was increased more than 700 times, but was not proportional to the area of the interface when different agents were used. Experiments were made on other reactions between immiscible liquids; in each case the emulsified system reacted more quickly than the corresponding unemulsified but agitated system.<sup>14</sup>

The penetrability of fat liquor emulsions, important to the leather industry, is discussed by Stiansy and Arnoldi. 15, 16

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    Barbieri, U. S. 2,078,555 (1939).
    Ayres, J. Ind. & Eng. Chem. 21, 902 (1929).
    Bennett, J. Ind. & Eng. Chem. 22, 1256 (1930).
    I.G., Fr. 845,661 (1940).
    Aczwa, Cologne. Buil. Soc. Chim. 3, 501 (1936).
    Kobe, Ind. & Eng. Chem. 31, 262 (1939).
    King, J. Soc. Chem. Ind. 57, 431 (1930).
    Stiansy, Collegium p. 230 (1928).
    Acceptable Collegium p. 202 (1939).
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<sup>16</sup> Arnoldi, Collegium p. 292 (1928).

It may be found advantageous, in industry, to apply an oil or related substance in the form of an emulsion in the following instances: when the material occurs naturally as an emulsion; when applying oil in water containing mixtures in which, after elimination of the water, the oil must act as a water-repelling agent; when a comparatively small quantity of viscous oil is to be distributed over a large surface; when a highly viscous oil is to be used in cases where it is impossible to apply heat; when by using an emulsion a final product of special properties is obtained; when the large total surface of the emulsified oil is an advantage; and when the charge on the colloidal particle can be used for electro-deposition.<sup>17</sup>

Oil-water emulsions of high oil content are poor conductors of heat.<sup>18</sup>

Agricultural sprays are usually O/W emulsions. Those used for killing insects are known as insecticidal sprays; those for insect eggs are termed ovicidal sprays.

The most commonly used oils in agricultural sprays are mineral and coal tar oils and their derivatives. In certain special cases fixed or essential oils are employed. Since most plants are injured by these oils it is necessary to apply them in a highly diluted form. Dilution with a cheap medium is desirable from the economic standpoint. Water plus an emulsifying agent does this very nicely. This type of emulsion must be O/W so that it can be further diluted in the field, and so that the more active ingredient may remain in the internal phase.<sup>1</sup>

In 1868 an emulsion of cresylic acid, soap and water was first described.<sup>2</sup> In chronological order the following agricultural emulsions were made known: kerosene and whale oil

<sup>17</sup> Eilers, Chem. Weekblad. 35, 129 (1938).

Pickering, J. Chem. Soc. 21, 2001 (1907).
 Woodman, J. Physical Chem. 33, 88 (1929).

<sup>&</sup>lt;sup>2</sup>T. A. Gardeners Monthly (Eng.) p. 11 (Jan. 1868).

soap; \* kerosene and soft or hard soap; \* kerosene and milk; \* heavy tar-oil. 6

Compound oil sprays containing fungicides introduce the problem of compatibility, e.g., emulsions containing Bordeaux mixture; nicotine; nicotine sulfate; lead arsenate; polysulfides; miscellaneous sprays.

These emulsions are made with cresol soap; 18 sodium resinate; 14 creosote and caustic soda; 15 carbon tetrachloride and cresol soap; 16 tar distillate and rosin soap; 17 fatty acids and glue; 18 pyrethrum extract. 10, 20

Weed killers, of the W/O type, are intended to kill wherever they contact plant life.<sup>21-24</sup> All other agricultural emulsions are O/W.

# Asphalt Emulsions 1

Bentonite is widely used in the manufacture of asphalt emulsions, which should be more correctly described as "aqueous dispersions" of asphalt.

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<sup>3</sup> Cruickshank, Ibid. 45, (Feb. 1875).
4 Cook, Mich. Agric. Exp. Sta. Bull. No. 58 (1890).
<sup>5</sup> Lodeman, The Spraying of Plants, p. 153 (1902).
<sup>6</sup> Del Gueres, F. Pflanzenkrakh 4, 4, (1894).
7 P.C.
<sup>8</sup> Ginsburg, J. Econ. Entom. 26,566 (1933).
9 Headlee, N. J. Agric. Exp. Sta. Bull. 469 (1929).
<sup>10</sup> Newcomer, U. S. Dept. Agric. Tech. Bull. 281 (1932).
<sup>11</sup> Batchelder, J. Econ. Entom. 24, 1152 (1931).
12 Tower, U. S. 1,869,562 (1932).
18 Woodman, J. Pomol. Hort. Sci. 4, 78 (1925).
<sup>14</sup> Penny, J. Econ. Entom. 14, 428 (1921).
15 Agric. Gaz. N.S.W. 35, 290 (1924).
<sup>16</sup> Takashima, J. Pub. Hlth. Japan. 3, 1 (1927).
<sup>17</sup> Hartzell, N. Y. Agric. Exp. Sta. Bull. 636 (1933).
18 Staniland, Long Ashton Ann, Report p. 78 (1926).
<sup>19</sup> Lodeman, The Spraying of Plants, p. 78 (1902).
<sup>20</sup> Ginsburg, J. Econ. Entom. 28, 599 (1932).
<sup>21</sup> Pipal, Indiana State Bull. 176 (1914).
<sup>22</sup> Woodman, Chem. News. 144, 21 (1932).
23 Woodman, J.S.C.I. 60, 91T (1931).
<sup>24</sup> Johnson, Calif. Agric. Exp. Sta. Bull. 528 (1932).
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<sup>1</sup> Amer. Colloid Corp., Chicago, Ill.

The usual commercial process <sup>2</sup> is to make a bentonitewater paste or gel, which should be as thick as the stirring or mixing mechanism will permit. This will be about 10 to 12 parts bentonite to 90 parts water. A stiff bentonite paste is desirable in mixing the emulsion, since it exerts a tearing action against the natural tendency of the asphalt to coalesce.

The bentonite-water gel is heated and the hot asphalt poured into it in a thin stream, with continuous stirring. After thorough mixing, the thick product can be further diluted with water, depending on its ultimate use. The final proportions of a fairly thick emulsion could be 10 parts bentonite, 90 parts water and 250 parts asphalt. For asphalt paint, 150 parts water with the above quantities of bentonite and asphalt would be effective.

When the compound has been formed, the bentonite surrounds the asphalt globules, forming a protective sheath against coalescence. The mixture remains plastic when kept in a container, and need not be heated to be applied to a surface. While in the plastic or mobile condition the volume of the hydrated bentonite, due to its swelling properties, is about seven times that of its dry volume. When the material is spread upon a surface the water evaporates, and the bentonite shrinks back to its original dry volume, forming cells of microscopic size between the particles of asphalt. These cells, now dry, are porous and become permeated by the asphalt, which gradually reaches complete coalescence. The film or body is then impervious to water.

The pH value of the final product is an important factor influencing stability. A slightly acid reaction is conducive to maintaining good adherence between the bentonite particles and the asphalt. Bentonite itself gives a slightly alkaline reaction in solutions and aluminum sulphate or small quan-

<sup>&</sup>lt;sup>2</sup> Miscall, Chem. & Met. Eng. 37 (1930); Kirchbraun, U. S. 1,918,759 (1933).

tities of acids are sometimes added to the water to lower the pH. e.g., 0.007% citric acid.

To improve suspendability, small amounts of other colloids may be helpful. Gum karaya, gelatin, starch, dextrin may be used for this purpose. One patented process mentions the use of a citric or tartaric acid to improve mobility, allowing for fluid emulsions containing less water and more asphalt. Another mentions that soap-asphalt and bentonite-asphalt emulsions be made separately and then mixed. It is said that bentonite emulsions may be more successfully made with high melting point bitumens, than with the low melting ones; whereas soaps act oppositely in this respect.

There are many patents describing and covering processes and refinements. A partial list follows:

### U.S.

1,302,810	1,663,652	1,734,437	1,788,706	1,918,759
1,479,042-43	1,679,475	1,738,509	1,802,561	1,948,881
1,498,387	1,690,020	1,738,776	1,829,722	1,957,408
1,517,075	1,691,765-6-7-8	1,738,906	1,865,634	1,959,586
1,615,303	1,722,431-2-3-4	1,781,654	1,882,834	1,960,112
1,615,303 1,620,899-900 1,663,095	1,722,431-2-3-4 1,725,645-6-7-8 1,733,493-4-5-6-7-8	1,781,654 1,782,536 1,787,418	1,882,834 1,889,437 1,916,885	1,960,112 1,963,231

Whereas some Venezuelan and Mexican asphalts are directly emulsifiable in aqueous alkali (presumably because they contain traces of naphthenic acids).<sup>2n</sup> most asphalts require a soap or another dispersing agent for satisfactory emulsification.

When the melted bitumen is emulsified by stirring into it a hot concentrated aqueous soap solution, the latter is considered desirable.

A highly important factor in production is the use of soft water. Hard waters must be softened, e.g., by the "Permutit" or other process, before use. Viscosity is a particu-

Preaubert, Brit. 9,422 (1905).

larly important property of asphalt emulsions for two reasons.<sup>2</sup> First, it governs the workability of the emulsion, which must be applicable to paving aggregate or other surfaces. For this purpose the viscosity must be low. Secondly, viscosity influences adhesion and it must be high enough so that the emulsion clings until phase separation results.

Bituminous emulsions made with soluble emulsifying agents, such as soap, are rendered fluid by the addition of substances, such as calcium chloride, which form insoluble compounds with the emulsifying agent. The drying or settling characteristics of such fluid emulsions are increased by the addition of stabilizers, such as sodium phosphate, which do not increase their viscosity.<sup>2b</sup>

Road emulsions contain 56–60% asphaltic bitumen, but emulsions can be successfully made with over 80% bitumen. The higher surface tension and specific gravity of tar make it more difficult to emulsify, but these disadvantages are overcome by the emulsifying tendencies of some of its constituents.

The degree of dispersion of a bituminous emulsion varies with the pH of the aqueous phase, and the maximum dispersion corresponds to a maximum stability; the stability is greatly affected by different emulsifiers, e.g., the stability of a Panuco bitumen without an emulsifier or softener lies within the pH limits of 10.2 to 12.5. When emulsified with 0.9% of olein the stability limit is found within a pH range of 8.1 and 12.6. Viscosity and breaking point are also affected by the pH. Control of viscosity by pH has some practical difficulties and it is wise to employ such materials as stearic acid or sulfonic acids.<sup>3</sup>

In actual use, the emulsifier must be present in as small a

<sup>&</sup>lt;sup>2</sup> P.C.

<sup>&</sup>lt;sup>2b</sup> B. I. Inc., Fr. 760,544 (1934).

<sup>&</sup>lt;sup>3</sup> Yellinger, 11e Congr. Mond. Petrole 2, 659 (1930).

proportion as possible, partially to avoid re-emulsification under traffic, and partially to avoid diminishing the adhesive power of the bitumen to the mineral matter of the road surface. As a rule, the more finely divided the binding material, the lower is the amount of emulsifier required. Generally, dissolved salts, both in the manufacture and in breaking on the road, effect the electric charges of the suspended particles to a degree sufficient to reverse or even destroy the colloid system.4

Another important property is emulsion stability, since the emulsion must not break until after application. A sensitive emulsion will break if the mixer runs too fast, or sometimes merely from gravity flow if the mixer is much higher than the receiving tank. On the other hand, asphalt emulsions may be made so stable that they can be centrifuged without breaking. Tall oil is an effective stabilizer, containing both resin acids and fatty acids. As a rule an asphalt which is difficult to emulsify gives sensitive emulsions, 5 requiring more stabilization than would a readily emulsifiable asphalt.

The most common bituminous emulsions used in road building are made with asphaltic bitumen, a residual petroleum refining pitch. Since their viscosity directly affects spraying and coverage, this property is quite important.1 Uniformity and globule size of bitumen emulsions are governed by the grade of bitumen used. Since there is a definite variation in every batch of bitumen, it is necessary to regulate the manufacturing procedure in order to maintain fairly uniform production.2

While colloid mills are preferred, other mixers may be used in most cases. To get optimal results, experimental runs

<sup>\*</sup> Spielmann, Munic. Eng. Sanit. Record 84, 624 (1929).

Nussel, Bitumen 11, 45 (1941).
 Jap. J. Eng. Abstracts 17, 7 (1939).

<sup>&</sup>lt;sup>2</sup> Khim. Ref. Zhur. 11, 119 (1939).

must be made to determine the best operating conditions.

Bitumen contains acidic materials which saponify with alkalies to form emulsifiers. Where the acidic content is too low, fatty acid or soap is added. Some free alkali is always needed to get the best dispersion.<sup>3, 4</sup>

The best procedure is to add the melted bitumen slowly to the aqueous alkali solution which is heated to a boil in the mixer. Emulsification is complete in 2–3 minutes. At this time some soap or other stabilizer is added if the emulsion is not used at once.<sup>5</sup>

In another method bitumen is melted with fatty acid in the mixer, and then a boiling concentrated aqueous alkali solution is added. This is then diluted with hot water.<sup>6</sup> The significance of various emulsifiers for bitumens is discussed by Rick.<sup>7</sup>

Silica, iron oxide and gypsum promote the formation of asphalt in water emulsions. Limestone, magnesium carbonate, calcium hydroxide, magnesium hydroxide and Portland cement promote water in asphalt emulsions.<sup>8</sup>

Becker "gives detailed directions for the laboratory preparation, commercial manufacture and testing of bituminous emulsions. A description of laboratory and technical equipment is also given with the aid of numerous photographs, photomicrographs, diagrams and tables. These tables summarize the results of laboratory tests of the preparation and properties of various emulsified asphalts containing such emulsifiers as tall oil, chlorinated tall oil, montan wax, naphthenic acid, olein and stearin pitch. The optimal proportions

<sup>3</sup> Kozi Doi. Jap. 129,057 (1939).

<sup>&</sup>lt;sup>4</sup> Technical Aspects of Emulsions, p. 136.

<sup>&</sup>lt;sup>5</sup> Montgomerie, Brit. 226,032.

<sup>&</sup>lt;sup>6</sup> MacKay, Brit. 202,021.

<sup>&</sup>lt;sup>7</sup> Rick, Teer v. Bitumen *32*, 113 (1934). <sup>8</sup> Ebberts, Rock Products *33*, 53 (1930).

<sup>&</sup>lt;sup>9</sup> Becker, Bitumen 11, 38 (1941).

and properties of asphalt, water and emulsifier have to be determined for each technical use by systematic series of tests.

Bitumen emulsions are classified <sup>10</sup> as to stability by Neubronner.

The use of asphalt emulsions for road building and water proofing of paper, concrete, and similar purposes from 1907–1928 is exhaustively reviewed by Leemans.<sup>11</sup>

### Cosmetic Emulsions

Emulsions figure predominantly in cosmetic preparations. The following are typical examples: cold cream, vanishing cream, shaving cream, hand lotion, bath-milk, etc. The first is usually of the W/O type, while the others are O/W.

The various aliphatic alcohol bases suitable for use in cosmetics and toilet preparations, comprise cetyl, cetylstearyl alcohol mixtures, technical stearyl, myristyl and oleyl alcohols and various proprietary agents based on admixtures of these with a small percentage of aliphatic alcohol sulfonates, such as sodium lauryl sulfate. Sulfonated aliphatic alcohols are produced by the reaction of sulfuric acid with alcohols of high molecular weight to give alcohol sulfates. Since sulfuric acid is dibasic, one acid hydrogen remains after esterification, this being replaced, for example, by sodium to yield the sodium salt. In the manufacture of cosmetics and toilet preparations, aliphatic alcohol sulfates are employed as emulsifying agents, soap substitutes and foaming agents. The most commonly used emulsifying agents consist of mixed aliphatic alcohols (about 90%) and aliphatic alcohol sulfates. Recently, phosphates, as well as sulfates, have been used. Such emulsifying agents are readily

<sup>&</sup>lt;sup>10</sup>Neubronner, Teer v. Bitumen 32, 211 (1934).

<sup>&</sup>lt;sup>11</sup> Leemans, Chem. Weekblad 26, 2 (1929).

self-emulsifiable in water, and give creams of the oil-in-water type, such as skin foods, nourishing cream, night creams. On the other hand, they can also form oil-in-water emulsions with a variety of oils, fats, and waxes.1

Some of the more recently introduced emulsifying agents, used in the cosmetic industry are:

"Acimul" Aminoalcohols Ammonium stearate, anhydrous Monostearin "Deramin" Diglycol Laurate "Emulgor" A Glyceryl Monostearate "Glycosterin"

Mannitol Oleate Monoethanolamine Sorbitol Laurate Triethanolamine "Trigamine" "Trigamine" Stearate "Trikalin"

# Errors in Making Creams 2

When using white oils, paraffin, petroleum jelly, stearic acids, etc., in the manufacture of creams, it must always be remembered that the qualities offered vary considerably. There are heavy and light mineral oils, paraffins of varying melting points, and petroleum jellies of varying viscosity. The quantity of water, for instance, which is to be added to hair creams, depends largely on the viscosity of the mineral oil used. It is preferable to use a heavy oil and more water rather than a thin oil and less water. Furthermore, the creams made with heavy oils are usually better and considered preferable by the buyer to those made of thin light mineral oils.

In making face creams the mistake is often made of not working at the correct temperatures. While a cream containing lanolin or similar absorption bases is made at slightly above room temperature, it is necessary in certain instances

<sup>&</sup>lt;sup>1</sup> Jannaway, Perf. Essent. Oil Record 30, 45 (1939). <sup>2</sup> Schimmel, Amer. Perf. 32 (Oct. 1941).

to increase the temperature of production. For instance, a higher temperature is required when it is a case of a mixture of paraffin with an emulsion containing soap, or if another emulsifying agent is concerned which is not particularly sensitive to heat and is used together with ceresin or ozocerite. Otherwise it is possible that the distribution of the added wax or paraffin is not sufficiently thorough.

If creams deteriorate occasionally, the reason for this may be that the materials used are not mutually compatible. For instance, the use of lemon juice in an alkaline cream is rather dangerous and often unsuccessful. Another reason for deterioration may be that distribution in the inner phase of the emulsion is at fault.

Another matter for consideration is that the presence of the electrolytes such as salts of magnesium, zinc, or bismuth, deficient viscosity, and the use of unsuitable emulsifying agents can considerably affect the quality of the cream. Occasionally insufficient emulsification may be corrected by passing the cream through a colloid mill or homogenizer. In certain instances the stability of the emulsion will thereby be increased and also the quality of the cream be improved. In some cases the stability of the cream may also be bettered by the addition of small quantities of wax and stearic acids. Certain emulsions such as creams containing gums and tragacanth are preferably made without the use of a homogenizer, as the emulsifying properties of the gum or tragacanth may be destroyed. The increased temperature and pressure caused by the passage through the homogenizer may furthermore have a detrimental effect on the already existing emulsion which may be sensitive to heat.

Another mistake which occasionally occurs in cream manufacturing is the simultaneous preparation and use of different types of emulsions. For instance the error may be made of adding comparatively large percentages of lanolin to a beauty

milk, based on triethanolamine stearate. Through the addition and the ensuing slow increase of water content, a water-in-oil emulsion will first be formed, which will then be changed into an oil-in-water emulsion with the result that the emulsion is unstable.

# Miscible, "Soluble" or Cutting Oils

A miscible oil is a clear solution of an emulsifier in an oil which emulsifies on mixing with water. A cutting oil is a "soluble" oil having lubricating, cooling, and non-corrosive properties.

In making miscible oils a coupling agent or mutual solvent is included to give a clear, uniform solution.

Free oleic acid is necessary to make clear water-miscible "soluble" oils, however it has a tendency to break emulsions made with a "soluble" oil and its relative proportion is often highly critical. The kind and quality of the mineral oil used has very little effect on the quantity of oleic acid used. Alcohol is usually added. It first acts as a liquefier and in large amounts, causes an increase in the quantity of oleic acid required. It exerts no direct effect on the emulsion.

The addition of alkali to a "soluble" oil, containing acid sulfonated oil, in the absence of alcohol, first decreases then increases the amount of oleic acid needed; in the presence of sufficient alcohol, the more alkali present the less oleic needed. The more neutralized the sulfonated oil, the better 'emulsifier it is.

When soaps are used phenols are often employed as introfiers 2.3 as are amyl alcohol,4 ethyl alcohol,5 glycols 6 and

<sup>&</sup>lt;sup>1</sup> Hart, J. Ind. & Eng. Chem. 21, 85 (1929).

Woodman, J. Agric. Soc. 17, 44 (1927).
 Woodman, J. Pom. Hort. Sci. 5, 43 (1925).

<sup>\*</sup> Woodman, J. S. C. I. 49, 93T (1930).

Melander, Wash. Agric. Exp. Sta. Bull. 197, (1926).
 P.C.

glycol ethers.<sup>7</sup> Triethanolamine soaps and fatty acid esters of polyhydric alcohols containing at least one free hydroxyl group (e.g., glycol monolaurate) are also used. Many liquids which are soluble in both phases can act as introfiers and add stability. In many cases the addition of water is also necessary,<sup>8</sup> thus a miscible oil is a three or four component system.<sup>9</sup> The quantity balance of each compound is a critical factor in producing miscible oils.<sup>10, 11</sup>

The pH of "soluble" oils usually is between 7 and 9. This is due to hydrolysis of the emulsifier. If, however, the water used contains appreciable amounts of salts of strong acids and weak bases, the emulsion will have a lower pH.<sup>12</sup>

Cutting fluids are used both to cool and to lubricate. When lubrication is important it is generally recognized that fatty oils are superior to mineral oils. The evidence apparently proves that the fixed oils owe their superiority not to differences in viscosity or surface tension, but to residual valence or acidity which causes them to adhere more firmly to the metal than do the mineral oils. Mineral oils may have substances added to them which will, for some purposes, make them superior to lard oil. For many purposes, the addition of fatty acids, pine oil or sulfur derivatives to mineral oils will make it possible to obviate the use of expensive fixed oils.<sup>18</sup>

## Detergent Emulsions

A detergent acts by suspending foreign matter in a liquid or foam. It floats off the "dirt." This, of course, presup-

<sup>7</sup> P.C.

<sup>8</sup> P.C.

Technical Aspects of Emulsions p. 85 (1936).
 Woodman, J. S. C. I. 52, 185T (1933).

<sup>11</sup> Lehnes, Farber Z 156 (1904).

<sup>12</sup> Billings, Amer. Dyestuff. Rept. p. 26 (May 16, 1938).

<sup>13</sup> Bingham, U. S. Bureau of Stand. Tech. Paper 16 No. 204, 35 (1922).

poses a lowered surface tension. Where the dirt is enclosed or imbedded in oil, fat, and the like, an emulsifying action is necessary. Added alkalies, within certain limits are often helpful,<sup>5</sup> as is the addition of solvents.

Difficulty in removing mineral oil from wool with soap and soda, is attributed to the great adhesive power between the oil and wool resulting from the great oil-water interface tension. This tension may be reduced by the addition of long chain aliphatic alcohols (e.g., oleyl alcohol) or cetyl alcohol sulfate.1 The use of 6% oleyl alcohol seems to be optimal. Greater amounts hinder oil removal.2 Ease of removal of mineral oil from wool is dependent in the first place on the distribution of oil-soluble polar compounds between wool-oil and oil-water interfaces, secondly on the degree of oil-water interfacial tension and, thirdly on adhesion.<sup>3</sup> The optimum pH for scouring is 9.5–10.7.

Emulsification in hard water is accomplished with salts of both sulfated higher fatty alcohols and substituted aromatic sulfonated acids, soap, and sodium hexametaphosphate (Calgon).4

## Food Emulsions

Both natural and man made emulsions serve as foods in numerous instances. Natural emulsions are typified by cream and milk. Man made edible emulsions are exemplified by mayonnaise, salad dressing and shortening.

If the proportion of oil to egg or to emulsion already produced is kept below a certain maximum, a stable emulsion

<sup>&</sup>lt;sup>5</sup> Hannay, J. Soc. Dyers & Color. 50, 273 (1934).

<sup>&</sup>lt;sup>1</sup> Speckman, Trans. Far. Soc. 29, 358 (1933). <sup>2</sup> Tech. Aspects of Emulsions p. 104 (1936).

<sup>&</sup>lt;sup>3</sup> Ibid., p. 106. 4 Ibid., p. 107.

always results regardless of temperature or manner of beating. If the proportion of oil exceeds a certain minimal proportion, the egg or emulsion already produced always becomes dispersed in the oil and no permanent emulsion is formed. If proportions between the limits are used the formation of a permanent emulsion depends upon such variables as temperatures and method of beating. If the egg is previously diluted with vinegar the proportion of oil which can be emulsified is greatly increased during the first and second additions of oil, but with increase in viscosity the maximum ratio of oil to emulsion rapidly approaches the value obtained when egg is used alone.6

Mayonnaise is a typical edible emulsion. It consists of a dispersed phase (vegetable oil), a continuous phase (water), emulsifying agents (egg volk and mustard and vinegar) and/or other flavoring materials. Egg yolk is the most efficient edible emulsifying agent for O/W emulsions. phosphatide content (chiefly lecithin) tends to produce O/W emulsions, whereas its cholesterol tends to the opposite type.2, 3 Any change in proportions of lecithin and cholesterol will affect the stability or type of emulsion.\* This is probably why fresh egg yolk is more efficient than the frozen product in which the lecithin is partially hydrolyzed. (Lecithin always undergoes hydrolytic changes on aging and thus will vary in its action. Cholesterol is relatively stable.) 1

In mayonnaise manufacture, a pre-mix of all the oil, emulsified in a small part of the aqueous phase, is added to the rest of the aqueous phase. The great ratio of oil to water tends to make the W/O type, but the emulsifying agent pre-

<sup>6</sup> Mark, J. Home Econ. 13, 447 (1921).

<sup>&</sup>lt;sup>2</sup> Seifriz, Amer. J. Physiol. 56, 124 (1923). <sup>3</sup> Bhatnagar, J. Chem. Soc. 119, 1760 (1921).

<sup>4</sup> Corran, Biochem. J. 18, 1368 (1924).

<sup>&</sup>lt;sup>1</sup> Technical Aspects of Emulsions, p. 91

vents this. The reason for this method is that it produces a high viscosity which permits better mixing and the use of a smaller amount of emulsifier. Mayonnaise and certain other types of oil-rich emulsions are not homogenized because such treatment may break the emulsion.

Powdered mustard is used in mayonnaise to prevent formation of lime soaps, when hard water is used, with resultant W/O type formation.<sup>5</sup>

Careful adjustment of viscosity in W/O emulsions and use of proper emulsifiers (Palsgaard Emulsion Oil) ensures stability up to 100° C with resultant saving in the amount of fat used.<sup>7</sup>

Breaking of mayonnaise and dressing may be due to: (1) wrong proportions of oil, water and egg (best proportions are 78, 17, 5); (2) too rapid addition of oil; (3) wrong type of agitation; (4) effect of heat; (5) freezing; (6) alum (as in pickle relishes); (7) salt causes thickening; (8) starch (added) is hydrolized by acid; (9) bacterial decomposition; (10) evaporation of water; (11) vibration.<sup>8, 9</sup>

Shortening is a W/O emulsion of edible oil or fat used in baking. Many emulsifiers have been used, e.g., polymerized or oxidized glycerides of unsaturated fatty acids.<sup>10</sup>

The necessity for having a hydrophilic group and a lipophilic group, in an effective margarine emulsifier, indicates the desirability of polyglycerol as a component. Exhaustive esterification of the polyglycerol with a suitable fatty acid is the next step in making an active emulsifier. A phosphatide group, esterified once with hydroxyethyltrimethylammonium hydroxide, provides a potent hydrophilic group. A good emulsifier for margarine must be miscible with water and

<sup>&</sup>lt;sup>5</sup> Food manufacture, 1, 17 (1934).

<sup>&</sup>lt;sup>7</sup> Petersen, Fette u Seifen 46, 458 (1939).

<sup>8</sup> Gray, Oil Fat Ind. 4, 410 (1927).

<sup>&</sup>lt;sup>9</sup> Technical Aspects of Emulsions p. 96. <sup>10</sup> Schou, U. S. 2,220,976 (1940).

should be effective at 0.5% concentration in fat-water mixtures.<sup>11</sup>

#### Latex Emulsions

Rubber latex, coming from various species of plant, possesses different properties. Most of them are dispersions rather than emulsions.<sup>1a</sup> The percentage of rubber, in natural latex, usually is 35–40. A natural protein is present as a stabilizer.

Latex is concentrated by creaming, warming and removing the cream layer mechanically; also by Irish moss, 1, 2, 3 other colloids, 4 gum tragacanth, 5 pectin, 6 gelatin, 7 ammonium alginate, 8 agar, and casein. 9 Creaming is hastened by centrifuging, 10 low electrical potentials, 11 evaporation after addition of soap, 12 addition of alcohol. 13 Saponin, peculiarly, acts as a stabilizing agent. 14

Dry powders used in concentrating latex must first be wetted by an aqueous solution of the stabilizer.<sup>8a</sup>

Concentrated latex, having a larger percentage of protective colloid, is usually more stable than normal latex.

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<sup>11</sup> Palladina, Maslo. Zhirov. Delo 2, 30 (1939).
  <sup>18</sup> Technical Aspects of Emulsions p. 121.
  <sup>1</sup> Biochem. Z. 149, 525 (1924).
  <sup>2</sup> Brit. 226,440 (1924); U. S. 1,754,842 (1930).
  <sup>3</sup> K.D.P., Brit. 237,269 (1929); Fr. 686,821 (1929); Westcott, U. S.
1,754,535 (1930).
  4 Rossman, India Rubber World 87 (vi) 33, (1933); Ibid., 88 (iii) 31,
(1933).
  <sup>5</sup> Johnson, Brit. 286,527 (1927).
  <sup>6</sup> McGavack, U. S. 1,647,805 (1927); Pektinges, Ger. 560,259 (1923).
  <sup>7</sup> Banks, U. S. 1,755,279 (1920).

<sup>8</sup> General Rubber, Brit. 294,002 (1927).
  <sup>9</sup> Loomis, U. S. 1,816,018 (1932).
  <sup>10</sup> Westcott, U. S. 1,754,535 (1930).
  <sup>11</sup> McGarack, Brit. 344,647 (1929); Ibid., U. S. 1,921,575 (1933).
  <sup>12</sup> Technical Aspects of Emulsions p. 126. 

<sup>18</sup> Stevens, Brit. 415,133 (1933).
  <sup>14</sup> Teague, U. S. 1,772,647 (1930); Nikitin, U. S. 1,880,975 (1932).
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3a Ibid., p. 128.

Hevea latex becomes acid on standing and coagulates. Ammonia is usually added to prevent this. Such preserved latex may be coagulated with salts (aluminum sulfate) while still alkaline.<sup>2a</sup> Sodium silicofluoride, hydrophilic materials (e.g., alcohol, acetone, carbon-black, cement) will achieve the same effect. These materials probably act by absorbing some of the water.

The viscosity of aqueous rubber dispersions first increases linearly with dispersion of the water in the rubber, and then decreases with formation of a dispersion of rubber in water. With increasing plasticity of the rubber, the quantities of alkali needed as dispersing agent decrease, and the sizes of the dispersed particles are smaller, and lie within narrower limits. The viscosity of the dispersion for a given concentration varies with alkali concentration.<sup>15</sup>

#### Leather Emulsions

In the manufacture of leather dilute O/W emulsions, called fat liquors, are used extensively. The oils are triglycerides or mineral oils and the emulsifiers are soap or sulfonated oils.

The following emulsions are used in the manufacture of leather:

Oil with glue or gum, gum and starch, sodium glycerophosphate, neats-foot oil with aluminum hydroxide; oil with esters of sulfo-fatty acids and albumin or glue; oil with tertiary or quaternary amine salts; oil with methyl cellu-

<sup>&</sup>lt;sup>28</sup> Ibid., p. 125.

<sup>&</sup>lt;sup>15</sup> Dogadkin, Colloid J. (U.S.S.R.) 1, 447 (1935).

<sup>&</sup>lt;sup>1</sup> Rohm, Brit. 353,846 (1929).

<sup>&</sup>lt;sup>2</sup> Rohm, U. S. 1,981,363 (1932).

<sup>&</sup>lt;sup>3</sup> Rohm, U. S. 1,751,217 (1930).

<sup>&</sup>lt;sup>4</sup> Bohme, A. G. Brit. 313,966 (1928).

<sup>&</sup>lt;sup>5</sup> Somerville, U. S. 1,883,042 (1932).

lose; 6 oil with lecithin; 7 oil with residue from extracted soy-bean phosphatides; 8 and oil with bentonite.9

#### Medicinal Emulsions

Toxic doses of diphtheria and tetanus toxin may be safely injected subcutaneously if they are emulsified in a finely dispersed oil in water emulsion (5% olive oil in 0.2% solution of sodium oleate and brought to pH 8 with sodium carbonate).<sup>1</sup>

Improved methods for making pharmaceutical emulsions of turpentine, paraffin oil and other oils are given by Hall.<sup>2</sup>

The most commonly used medicinal emulsions are those of highly refined viscous mineral oil and cod-liver oil.

### Paint Emulsions

Most emulsion paints are O/W carrying pigment, wetted by water. The stabilizer is usually a protein (casein or glue) and the oil phase (varnish) is of high viscosity. The emulsifier may be soap or a sulfated product. The warm oil is usually added to the water containing the protein and soap. Emulsification proceeds smoothly on mixing and the water wetted pigment is dispersed in the aqueous phase. The emulsion may be slowly diluted with water before use.<sup>1</sup>

Breaking of paint emulsions often is due to shaking in transportation or too vigorous mixing before application with the brush. This is due to penetration of pigment particles into the protective oil layer and their being wetted by

<sup>6</sup> I.G., Ger. 524,211 (1926).

<sup>7</sup> Bollman, Ger. 414,399 (1927).

<sup>&</sup>lt;sup>8</sup> Muhlenwerke, Ger. 522,041 (1927).

<sup>&</sup>lt;sup>9</sup> Progil, Fr. 742,563 (1933).

<sup>&</sup>lt;sup>1</sup> Walsh & Frazer, Brit. Med. J. March 10, 1934.

<sup>&</sup>lt;sup>2</sup> Hall, Pharm. J. 135, 133 (1935).

<sup>&</sup>lt;sup>1</sup> Khim, Referat. Zhur. 8, 103 (1939).

the oil phase. Aging of the emulsion before pigmentation often improves stability.

Certain emulsion paints are as of good quality as non-emulsion paints; they are more economical; and have equal covering power. The W/O type is superior; the inclusion of oxidized instead of raw oils is preferable; and less than 25% water should be used. These emulsion paints can be used indoors or outdoors when properly made, but they are not recommended for unprimed iron.<sup>2</sup>

The amount of emulsifier determines the type of paint emulsion. Using ethanolamine or triethanolamine soap 0.25-6% gives W/O while 10% gives O/W.

Emulsions containing glue <sup>8</sup> may give rise to the following film types: (1) hydrophilic films with a glue-cellular structure, (2) two-layer films with a continuous oil surface and an undersurface interspersed with glue cell walls, and (3) films of true varnish type. Emulsions yielding type-1 films should be based on casein rather than on animal glue, which is the more easily reversed. Excess of alkali may be incorporated into emulsions containing saponifiable oils on account of the emulsifying action of the soaps produced. Emulsifiers such as sulfonated fatty alcohols or triethanolammonium esters should be added to the media containing unsaponifiable matter, and hydrophilic wetting agents to pigmented emulsions.

## Polish Emulsions

Emulsion shoe polishes consist of mixtures of waxes, with or without solvents, emulsified with alkalies. Potassium carbonate is preferred as an alkali because it gives more uniform dispersions. It is necessary to use only a small amount of alkali to saponify part of the fatty acid contained in the wax. This then acts as an emulsifier for the rest of

<sup>&</sup>lt;sup>2</sup> Ibid., 11, 109 (1939).

<sup>&</sup>lt;sup>3</sup> Wagner, Chem. Z. 60, 393 (1936).

the wax. Soap is often added as an auxiliary emulsifier because of its bodying and lubricating effect, and especially when using waxes containing little or no free fatty acid.

A typical procedure is to dissolve the soap, alkali, water and soluble dye in boiling water in the foregoing order and then add the melted waxes, resins and solvents slowly with good mixing. Heating is continued until all the carbon dioxide gas has been eliminated and until the emulsion is homogeneous. Containers are best filled at the lowest possible temperature (just above the setting point) and containers should be allowed to cool uniformly and not be disturbed until their contents have solidified.

Sodium carbonate gives harder products that will hold larger percentages of water. Potassium carbonate is preferred because the finished product, made with it, has a lesser tendency to shrink and contract. Borax, ammonia and organic amines are also used as alkalies to obtain special polishes.

Floor, furniture and automobile polishes are essentially very similar to shoe polishes except that they are more fluid. While formulation is different, their method of manufacture is the same.

# Transparent Emulsions

Transparent emulsions are made by equalizing the refractive indices of the phases <sup>1</sup> and stabilizing the emulsion by equalizing the phase densities.<sup>2</sup> Carbon tetrachloride and glycerol are suitable liquids for raising refractive indices.<sup>3</sup> Concentrated invert sugar and sucrose solutions may also be used for this purpose. These transparent emulsions can be made very stable by proper formulation and technique.

<sup>&</sup>lt;sup>1</sup> Rector, U. S. 1,389,161 (1921); Whatmough, U. S. 1,663,323 (1928). <sup>2</sup> Volek, U. S. 1,914,902 (1933); Imray, Brit. 200,036 (1923); Hartman, U. S. 1,611,190 (1826).

<sup>&</sup>lt;sup>8</sup> Holmes, Introductory Colloid Chemistry p. 82, (1934).

Coconut oil ethyl ester is suitable for lowering refractive indices of oil.

Glycerin and tartaric acid are used for increasing refractive indices of sugar solutions.

Gelatin is the most efficient of the peptizing agents examined, while gum arabic ranks second. Examples of these are the transparent emulsions of orange, peppermint, lemon, lime and rose oils made with gelatin, gum arabic, gum tragacanth or agar-agar as emulsifier.

Agar-agar and tragacanth have sometimes proved unsatisfactory peptizing agents.<sup>4</sup> The problem of graining may be solved by employing a mixture of two parts invert sugar and one part sucrose, instead of straight invert sugar in the dispersion medium. The colloid mill is indispensable in the preparation of this type of dispersion.

Emulsions of varying colors are made with nitrocellulose (dry) glycerol, and carbon bisulfide added in a definite way, in different proportions.

These colors are also changed by heat.<sup>5</sup> To make similar chromatic emulsions, the continuous phase must have two mutually soluble liquids, one having a high refractive index and high optical dispersive power. Benzol, rubber and water also produce chromatic emulsions.

# Wax Emulsions

Stable wax emulsions, usually have a uniform particle size of about 10<sup>-4</sup> cm.

Most troubles in making wax emulsions are caused by specks or clumps of incompletely melted or chilled wax. It is therefore, essential that a sufficiently high temperature of both phases be maintained to avoid such difficulties.<sup>6</sup>

<sup>&</sup>lt;sup>4</sup> Whitmore, J. Ind. & Eng. Chem. 21, 878 (1922).

Holmes, J. Amer. Chem. Soc. 21, 878, (1929).
 Billings, Amer. Dyestuff Rept. May 16, (1938).

## CHAPTER VI

# DISPERSING AGENTS AND WETTING AGENTS

# Dispersing Agents

A dispersing agent is a compound which separates aggregations of particles of matter by neutralizing the cohesive attraction between them. A dispersing agent may have but little effect on the surface tension. Consequently, its wetting properties may be negligible. Its action is very specific—working well in some systems and not in others. A dispersing agent may be either inorganic or organic. An interfacial tension against mineral oils below 11 dynes per c.c. usually indicates a good dispersing agent.

A dispersing agent shows the greatest effect with highly flocculated hydrophobic systems. In very adhesive products, e.g., coagulated latex, mechanical force is often necessary before the dispersing agent can exert its action.

Protective colloids should not be used with dispersing agents as they interfere with each other, because of their adsorptive properties. This caution also applies to using wetting agents with dispersing agents.

The following are a few materials that are being used commercially as dispersing agents:

<sup>&</sup>lt;sup>1</sup> Daniel, Rubber Age 48, (Dec. 1, 1939).

Darvan; <sup>1a</sup> Daxad <sup>2</sup>
"Glaurin" <sup>3</sup>
Glycol Naphthenate <sup>3</sup>
"Hornkem" <sup>4</sup>
Sodium Lignin Sulfonate <sup>5</sup>

# Wetting Agents

Wetting agents are generally materials which, in small amount, greatly reduce the surface tension of water and lower the interfacial tension between water and another contacting surface. A good wetting agent shows a surface tension of 30 dynes per c.c. or less in solution. Interfacial tension is the surface tension at the interface (region of contact) between two immiscible phases. This region may be liquid and air, liquid and a solid, or two liquids.

There seems to be considerable confusion as regards the terms "wetting" and "dispersing." As stated previously, a wetting agent is a material which will reduce the surface tension of water and lower the interfacial tension between the water and another surface, so as to form a solid-liquid, or liquid-liquid interface. On the other hand, a true dispersing agent usually has little effect on the surface tension of water. An aqueous dispersing agent is a material which promotes the separation or deflocculation of particles, which is accomplished by overcoming the adhesive forces between the individual particles.<sup>1</sup>

Wetting agents usually contain, both polar and non-polar groups. The following table 11 gives their characteristics.

<sup>&</sup>lt;sup>1a</sup> R. T. Vanderbilt Co., New York, N. Y.

<sup>&</sup>lt;sup>2</sup> Dewey & Almy Chem. Co., Cambridge, Mass.

<sup>&</sup>lt;sup>3</sup> Glyco Products Co., Inc., Brooklyn, N. Y. <sup>4</sup> A. C. Horn Co., Long Island City, N. Y.

<sup>&</sup>lt;sup>5</sup> Marathon Chemical Co., Rothschild, Wisc.

 $<sup>^{1}\,\</sup>mathrm{Wetting}$  Agents, Their Structure, Etc.; Paper Trade Journal, August 1940.

<sup>&</sup>lt;sup>11</sup> Christmann, Amer. Cyanamide Co. Tech. Paper 17 (1930).

The polar group has an affinity for water and water-soluble materials and the non-polar group prefers oily and waterinsoluble materials. The balance of these two groups and their positions in the molecule determine the compound's characteristics.

#### POLAR-NONPOLAR COMPOUNDS

Polar (Salt)
Ionized
High Dielectric Constant
High Melting Point
Soluble in Water
Insoluble in Organic Solvents
High Boiling Point
Melt Conducts Electricity

Nonpolar (Mineral Oil)
Nonionized
Low Dielectric Constant
Low Melting Point
Insoluble in Water
Soluble in Organic Solvents
Low Boiling Point
Melt does not Conduct Electricity

Many varieties of wetting agents are available and used for different purposes under different conditions. The most commonly used method of evaluation is that of Draves-Clarkson.<sup>12</sup> It consists of measuring the time necessary for a 5-gram skein of unboiled two-ply cotton yarn to sink in a definite concentration of a wetting agent in water at a definite temperature.

Anion surface active compounds are characterized by having a negative charge on the surface active or non-polar group. On electrolysis of a solution of such a compound the latter group goes to the anode.

Cation active compounds are those whose cation is the effective agent. On electrolysis of a solution of such a compound, the active non-polar group travels to the cathode. Compounds in this class are the quaternary ammonium salts, sulfonium and phosphonium compounds.

Mixing anion and cation active compounds is detrimental

<sup>12</sup> Draves, Amer. Dyestuff Rept. 28, 425 (1939).

as they tend to form a precipitate by neutralizing their charges.<sup>13</sup>

Non-ion active compounds are those emulsifiers possessing inactive anions and cations, e.g., nonaethylene glycol laurate or "Emulgor A." <sup>14</sup>

As the length of the polar chains is reduced, the solubility is increased. It is noted that the sodium salt of dioctyl ester of sulfosuccinic acid is soluble to the extent of approximately 1.5% and that as the length of the polar chain is reduced still further, the solubility is further, though not markedly, increased. However, in doing so, the wetting power is adversely affected in ordinary water. For example, the sodium salt of the dibutyl ester of the sulphonated dicarboxylic acid has no wetting power in ordinary water at a concentration of 1%.15

Wetting intensity and protective colloid properties are unrelated for many wetting agents.

Wetting agents are specific. One agent may work well in the presence of a hydrophobic material and not with a hydrophilic material. Therefore, stating that a substance is a good wetting agent is incomplete unless the exact nature of the surface or interface is indicated.<sup>16</sup>

Some of the more commonly used wetting agents are the soluble salts of sulfated higher fatty alcohols, sulfonated amides, fatty acid ester sulfonates, sulfonated ethers, alkyl aryl sulfonates and polyhydroxy alcohols partially esterfied with higher fatty acids. A partial list of surface active agents follows:

<sup>&</sup>lt;sup>13</sup> Dhingra, J. Soc. Dyers Colourists 53, 91 (1937).

<sup>&</sup>lt;sup>14</sup> Glyco Products Co., Inc., Brooklyn, N. Y.

<sup>&</sup>lt;sup>15</sup> Wetting Agents, Their Structure, etc. Paper Trade Journal, Aug. 22, 1940.

<sup>16</sup> Bartell, J. Ind. Eng. Chem. 31, 31 (1939).

#### COMMERCIAL SURFACE-ACTIVE AGENTS

Produc	

Aerosol AY

Aerosol 1B

Aerosol MA

Aerosol OS Aerosol OT

Alco Penetrant L Alframine DCA Aliphatic ester sulfate Alkamine W Alkanol B, SA, HG.

Alkanol S

Amalgol

2-Amino-1-butanol

2-Amino-2-ethyl-1,3propanediol

2-Amino-2-methyl-3-hexanol

2-Amino-2-methyl-1,3-propanediol

2-Amino-2-methyl-1-propanol

Anthrapole Appretole

Arctic •Syntex A

Arctic Syntex M

Arctic Syntex T Areskap

Aresket

Aresklene

Composition

Diamyl ester of sodium sulfo-

succinic acid

Dibutyl ester of sodium sulfosuccinic acid

Succinic acid

Dihexyl ester of sodium sulfo-

succinic acid

Alkyl aryl sulfonate

Dioctyl ester of sodium sulfo-

succinic acid

Alkyl aryl sulfonate

Sulfated glycerylamide

Oil ester sulfate

Sodium alkyl naphthalene sul-

fonate

Sodium tetrahydronaphtha-

lene sulfonate

Fatty acid soaps

Sulfonated oil + solvents
Glyceryl sulforicinoleate
Oleic acid ester of sulfonated
aliphatic compound
Sulfate of mixed fatty acid
monoglycerides

C<sub>17</sub>H<sub>33</sub>CONH-C<sub>2</sub>H<sub>4</sub>-SO<sub>3</sub>Na

Monobutyl phenyl phenol sodium monosulfonate

Monobutyl diphenyl sodium monosulfonate

Dibutyl phenyl phenol sodium

disulfonate

$P_1$	rod	uc	ŧ

Aromine PM Arylene Avitex AD Bensapol Betasol OT-A

Bozeto1 Burkol C.A.C. 2720 Calgon Cominol 1

Cresol Cyclohexanol Daconol Darvan

Daxad Deceresol OT

Detanol

Diethylenetriamine Soaps Diglycol Laurate Dilex Dipex

Dreft Drene Duponol D. LS Duponol 80, WA, ME Dry PC Special WA 'Duponol G, OS, WS Elemite "Emulgor A"

Gardinol LS, WA, Special WA.

Glyceryl Monostearate

## Composition

Organic sulfonate + solvents Sulfonated aromatic Fatty alcohol sulfate Sulfonated oils & solvents Sulfonated ester of dicarboxylic acid Sulfonated castor oil derivative Organic sulfonate Phosphorated amine Sodium hexametaphosphate Derivative of higher sulfonated

alcohol  $C_6H_5OH-CH_3$ Hydrogenated phenol Alkyl aryl sodium sulfonate Polymerized organic salts of alkyl aryl sulfonic acids Same

Dioctyl ester of sodium sulfosuccinic acid Derivative of higher sulfonated alcohol

Purified sulfo-lignin Water-soluble sulfonated mineral oil Fatty alcohol sulfate Same Fatty alcohol sulfates

Same Same Sulfonated oils & solvents Polymerized ethylene glycol ester

Fatty alcohol sulfate

Halo

Hamico 169
Hartex Oil K-40
Hartex Oil L-33
Hartolein K-122
Hartopene
Hornkem 1
Hornkem 3
Hornkem 31
Hydroxyethyl ethylene
diamine Soap
Igepal C

Igepon AP Extra

Igepon T Integritol

Intramine

Invadine B.C.N.

Lamepon A Lamepon 4C Lamepon 10-N Lanitol P Lanitol S

Lanole B Lauramine Leonil SA

Loupene Loupole W-950 Maprofix Mapromin Mapromol Mercerol Composition

Sulfate of mixed fatty acid
monoglycerides
Sulfonated ester
Sulfated oils + solvents
Glycol soaps + alkali
Pine oil soap + alkali
Pine oil gel
Processed waste sulfite liquor
Hornkem 1 + wetting agents
Processed waste sulfite liquor

Polymerized ethylene oxide condensate
Oleic acid ester of long chain aliphatic
C<sub>17</sub>H<sub>33</sub>CONHC<sub>2</sub>H<sub>4</sub>SO<sub>3</sub>Na
Mixed sulfonated oils, esters, alcohols
Sodium salt of sulfonated lauryl and myristyl collamide

Sodium alkyl phenylene sulfonate
Protein fatty acid condensate
Same
Protein fatty acid condensate
Sodium alkyl aryl sulfonate
Sodium alkyl aryl sulfonate +
alkali
Sodium oleate + solvents

Naphthalene sulfonic acid derivative Sulfonated oils & solvents Sulfonated oils & solvents Sulfated fatty alcohol Same Same

Merpentine

Merpol B.C.

Methylcyclohexanol

Modinal D, ES

Monostearin Monosulph

Naccolene F

Nacconol E

Nacconol EP

Nacconol NR

Nacconol NRNO

Nacconol NRSF

Naccosol A

Nekal A, BX

Neomerpin N Neomerpin VD

Nonaethyleneglycol oleate

Nopco 1935

Nopco 1967

Nopco DID

Novonacco

Novonacco NN

N.S.A.E.

Nusope 33A, P50

Oleo Glyceryl Sulfate C, O

Onyxol 66

Orthocen

Orvus

Para Oil

<sup>2</sup>emeko

netrolin AC

Composition

Sodium alkyl naphthalene sul-

fonate

Alcohol sulfate

Fatty alcohol sulfate

Glyceryl monostearate, special

Sulfonated castor oil

Modified alkyl aryl sulfonate

Sodium alkyl aryl sulfonate

Same

Same

Same Same

Sodium alkyl naphthalene sul-

fonate

Sodium alkyl naphthalene sul-

fonate

Alkyl naphthalene sulfonic acid

Sodium alkyl naphthalene sul-

fonate

Sulfonated oil, vegetable type

Sulfonated alkyl hydrocarbon

Sulfonated fatty amine

Modified sodium alkyl naph-

thalene sulfonate

Modified alkyl naphthalene sul-

fonic acid

Alkyl naphthalene sulfonic acid

Alkali soap of naphthenic acids Oil ester sulfate

Same

Sulfonated higher phenol

Fatty alcohol sulfate

Saponified oils

Sulfated alcohol

Sodium salt of alkylated naph-

thalene sulfonic acid

## Pentogen

Phi-\$\phi\$-Sol
Phi-\$\phi\$-Sol W.A.
Pineol
Pyridine
Quadrafos
Quaker 3-X
Quaker 700-X
Quaker Dianol D

Quaker Pro-So-Tex 55 Quaker Pro-So-Tex 58

Quaker Pro-So-Tex 75 Quix Quixite Radiactine Rapidole H

Royox Sandozol G Sanol 1, S, T Santomerse Sapamine A

# Soapotol

Solvadine NC Conc. Stablex A Stablex G Sulfanole K

Sulfanole PB
Sulfatate
Sulfonated Luxolene
Supergel T.B.
Supersulfate F.S. Powder

# Composition

Sulfated compound containing solvents
Sulfonated oil ester
Same
Sulfonated oil + terpenes

Sodium tetraphosphate
Sulfonated pine oil
Sulfonated cresylic compound
Combination sulfonated higher
alcohol & aldehyde condensates

Aromatic sodium sulfonate Sulfonated petroleum derivative

Alkylated aryl sulfonate
Sulfonated fatty acid
Sulfonated fatty acid
Algin soap compound
Saponified sulfonated vegetable oils + solvents
Pine oil with soap
Sulfonated fat
Sulfonated aryl alcohols
Alkylated aryl sulfonate
Diethyl aminoethyl oleyl amide
acetate
Sulfonate derivative of higher

Sulfonate derivative of higher alcohol

Alkylated aryl sulfonate Naphthalene sulfonate Petroleum sulfonate

Fatty amide sodium sulfonate + solvents

Fatty amide sodium sulfonate Sulfonated hydrocarbon Fatty ester sulfonate Soluble terpene sulfonate

Sulfated aryl alcohol

Surpasol

Teel
Tenesol A, B, C, D, F concentrates.
Tensol

Tergitol Penetrant 4.7

Tergitol Penetrant 0S

Tergitol Penetrant 4T

Tetraethylene Pentamine Soaps Tetranol 1638 Tinapol Oil NE Tris (hydroxymethyl) aminomethane Soap Triton 720 Triton 812

Triton E-79
Triton K-12
Triton K-60
Triton NE
Triton W-30
Twitchell Base 262

Triton E-40

Twitchell Base 265 Twitchell Oil 3X Ultravon K, W

Ultrawet 40, 4D (+ sodium sulfate) Ultroil Unitex Vel Composition
Mixture of sulfonated oils,
esters, alcohol
Fatty alcohol sulfate

Sulfonated ethers
Same
Sodium salt of higher secondary alkyl sulfate
Sodium salt of higher primary alkyl sulfate
Amine salt of higher secondary alkyl sulfate

Sulfonated fatty ester Sulfonated ester

Sulfonated ether
Condensed sulfonated ether
Salt of sulfonated and sulfated
ether
Amine salt of sulfated ether
Quaternary ammonium salt

Same
Organic polyether alcohol
Sulfated aromatic ether alcohol
Sodium sulfonates of mineral
oil

Same
Same
Sulfonated complex stearyl
alkyl compound

Sulfonated vegetable oil
Alkylated aromatic sulfonate
Sulfate of mixed fatty acid
monoglycerides

Aromatic monosodium sulfo-

P	'n	'n	d	11.	c	ŧ.

W. A. 35A, B, C

W. A. 58

Warcosol

Warcosol 897

Wetanol

Wetonite
Wetsit
Xynomines
Yelkin C
Yelkin T

Yelkin T-X15

# Composition

Phosphorated higher alcohol

 $(\text{octyl})_5 \text{Na}_5 \text{P}_6 \text{O}_{20}$ 

Phosphorated Higher alcohol

 $(capryl)_5 Na_5 P_6 O_{20}$ 

Alkyl naphthalene sodium sul-

fonate

Hydrocarbon sodium sulfo-

nate + solvent

Modified sulfated fatty acid

ester

Sulfonated ester

Alkylated aromatic sulfonate Sulfated boro-fatty acid amide Water-dispersible lecithin

65-70% lecithin 30-35% soy-

bean oil

Lecithin compound

### CHAPTER VII

# LIST OF EMULSIFYING AGENTS

```
Abrocol (Glyceryl monostearate)<sup>1</sup>
  Acimul (A modified glyceryl monostearate used with weak
acids)2
  Agar 8
  Agar, pectin and gelatin 4
  Albasol (Sulfonated fats)<sup>5</sup>
  Albatex BD (Sodium m-nitrobenzene sulfonate)6
  Albumin 7
  Albumol (Complex albuminoid)<sup>8</sup>
  Aldol urea reaction products 9
  Algin 10
  Alginic acid or sodium alginate 11
  Alkyl or aromatic esters of petroleum sulfonates 12
  Alkyl guanidine 12a
  Alkyl sulfates of high molecular weight 18
  Alkylaminoethane sulfonic acids 14
  Alkylene oxides, polymerized 15
  Alkylolamine esters, polymerized (e.g., Polymerized trieth-
anolamine) 16
  Alronol 90 (Propylated amines) 17
  Alumina 18
  Alumina, activated gelatinous 19
  Alumina, powdered, or iron oxide gels 20
  Aluminum or calcium naphthenates 21
  Aluminum oxide, hydrated 22
  Aluminum palmitate 23
  Amide, acid 24
                              114
```

Amide, borated alkyl-25

Amide, castor oil 26

Amide, coconut oil 27

Amide, oleic 28

Amide, sperm oil 29

Amide, stear-30

Amide, stearyl monoglycol 31

Amide of substituted fatty acid 32

Amide, sulfated alkyl-33

Amide, wool fat 34

Amido fatty acid and sodium salts of their sulfates 35

Amine, amidostearyl diethylene-36

Amine, castor oil 37

Amine, coconut oil 38

Amine, dibutyl-39

Amine, di-2-ethylhexyl-40

Amine, di-2-hydroxyethyl-41

Amine, dioctyl-42

Amine, ethylenedi-48

Amine, monoamyl-44

Amine, monobutyl-45

Amine, oleic 46

Amine, tributyl-47

Amine, triethanol 48

Amine, tri-2-hydroxyethyl 49

Amine, triisopropanol 50

Amino-alcohol and synthetic resin acid reaction product '

Amino alkyl phosphatidic acids 52

2-Amino-2-ethyl-1, 3-propanediol 53

2-Amino-3-hexanol 54

2-Amino-2-isopropyl-1, 3-propanediol 55

2-Amino-2-methyl-1-butanol 56

2-Amino-2-methyl-3-hexanol 57

2-Amino-2-methyl-1, 3-propanediol 58

2-Amino-2-methyl-1, 3-propanediol oleate 59

2-Amino-2-methylol-1, 3-propanediol stearate 60

2-Amino-2-methyl-1-propanol 61

2-Amino-1-pentanol 62

2-Amino-2-propyl-1, 3-propanediol 63

3-Amino-4-heptanol 64

3-Amino-3-methyl-2-butanol 65

3-Amino-2-methyl-4-heptanol 66

3-Amino-3-methyl-4-heptanol 67

3-Amino-3-methyl-2-pentanol 68

Amino mineral oil sulfonate 69

5-Amino-4-octanol 70

Aminostearin (Modified amine soap)71

Ammonium laurate, anhydrous 72

Ammonium laurate, conc., S 73

Ammonium laurate S 74

Ammonium linoleate 75

Ammonium linoleate paste S 76

Ammonium oleate 77

Ammonium resinate 78

Ammonium salt of sulfonated shale oil 79

Ammonium salts, long chain quaternary, e.g., ammonium chloride and water soluble cellulose ethers <sup>80</sup>

Ammonium salt of phosphated castor oil 81

Ammonium shale oil sulfonate 82

Ammonium stearate, anhydrous 83

Ammonium stearate paste S 84

Ammonium sulfoicthyolate 85

Ammonium sulfoleate 86

Ammonium sulfonates 87

Amoa (Reaction product of caustic potash or casein) 88

Antimony trichloride 89

Aqualoid (Lecithin hydrate and 10% alcohol) 90

Aquamel (Animated hydrocarbon) 91

Aquaphil 92

Aquasol (Highly sulfonated castor oil)93

Arctic Syntex A (Oleic acid ester of an aliphatic compound sodium sulfonate)94

Arctic Syntex T (C<sub>17</sub>H<sub>33</sub>.CO.NCH<sub>3</sub>.C<sub>2</sub>H<sub>4</sub>SO<sub>2</sub>Na)<sup>95</sup>

"Areskap" (Neutral salt of sulfonated butyl diphenyl compound) 96

"Aresket" (Neutral salt of sulfonated butyl diphenyl compound) 97

"Aresklene" (Neutral salt of sulfonated butyl diphenyl compound) 98

Arkol base (Olive oil emulsifying base)99

Asphalt 100

Asphalt resin or petroleum tar 101

Barium oleate 102

Base 2210 (Naphthenic and petroleum sulfonate soap) 103

Betramine (Sulfonation product of a high molecular aliphatic aldehyde condensation) 104

Bile 105

Blendene 106

Blood albumin, casein or other protein, hydrolyzed and treated with alkali  $^{107}$ 

Blood serum with or without lecithin 108

n-Butylamine 109

Caesium oleate 110

Calcium carbonate, calcium arsenate, lead arsenate, zinc oxychloride, basic zinc chloride, Oxford clay, ferrous hydrosulfide or glycerin 111

Calcium ceryl sulfonate 112

Calcium chloride and borax 113

Calcium ricinoleate 114

Calcium triricolein phosphite 115

Carasol (Sulfonated oil)116

Carbitol citrate 117

Carbon black 118

Carbosterol (Carbitol stearate) 119

Carob bean extract and alkali 120

Casein 121

Casein, alkaline, with soap 122

Cellulose, alkylated 123

Ceryl potassium sulfate 124

Cetyl acid sulfate 125

Cetyl alcohol with sodium lauryl sulfate 126

Cetyl dimethylaminoacetate hydrobromide 127

Cetyl or stearyl alcohols 128

Cetyl or stearyl alcohol with resin alcohol and water 129

Chlorinated fatty acids with strong alkalies 180

Chlorinated oil soluble petroleum sulfonate soap 131

Cholestenone sulfonate salts 132

Cholesterol 133

Cholesterol betaine hydrochloride 134

Cholesterol, isocholesterol, metacholesterol, oxycholesterol, lanolin alcohol, ceryl alcohol, carnaubyl alcohol, cetyl alcohol and lanolin 1835

Cholesterol laurate 136

Cholesterol or oxycholesterol and albumins 137

Cholesterol stearate 138

Clay, colloidal, with calcium silicate 139

Coal, brown, or peat 140

Colloresin (Methyl cellulose)141

Condensation product of aminoguanidine with higher fatty acids 142

Condensation product of 1-chloronaphthalene with formaldehyde 148

Condensation product of  $\alpha$ -diethylamino- $\beta$ -hydroxypropylamine with oleic or stearic acid;  $\alpha$ -diethylamino- $\beta$ -hydroxy- $\alpha$ -phenylaminopropane with oleic or stearic acid chloride 144

Condensation product of fatty acid halides and albumin decomposition products 145

Condensation product of oleic acid and ethylene oxide 146

Condensation product of secondary or tertiary hydroxyalkylamines and aromatic acids, e.g., triethanolamine benzoate 147

Condensation products of starch, dextrin or cellulose with stearic, palmitic, oleic or linoleic acid 148

Condensation product of sulfated mineral oil fraction with monohydric saturated alcohol 149

Copper resinate 150

Cresol linoleic or phenololeic acids 151

Cyclic urethans 152

Cyclono (Methyl cyclohexanone glyceryl acetal) 158

Cyclopon A and GA (Fatty acid amide alkylsulfonate) 154

Deceresol A Y (Sodium salt of diamylsulfosuccinate, U.S.P. 2,028,091)<sup>155</sup>

Deceresol M A (Sodium salt of dimethylamylsulfosuccinate, U.S.P. 2,028,091)<sup>156</sup>

Deceresol O S (Sodium salt of diisopropylnaphthalenesulfonic acid) 157

Deceresol OT (Sodium salt of dioctylsulfosuccinate, U.S.P. 2,028,091)<sup>158</sup>

Dicholesteryl phosphate 159

Diethanolamine naphthenate 160

Diethylaminoethanol 161

Diethylaminoethanol 162

Diethylene glycol glyceryl monooleate 163

Diethylene glycol glyceryl monoricinoleate 164

Diethylene glycol monolaurate (Diglycol laurate neutral)<sup>165</sup>

Diethylene glycol monooleate 166

Digitonin 167

Diglycol laurate S 168

Diglycol oleate 169

Dihydric alcohol completely esterified with a higher fatty acid and containing free fatty acid <sup>170</sup>

Dimethylcyclohexylamine oleate 171

Distearin (Glyceryl distearate)<sup>172</sup>

Dodecyl, cetyl or octadecyl pyridinium trialkylammonium halides <sup>178</sup>

Duponol G (Long chain alcohol sulfonate)174

Duponol OS (Oil soluble alkyl sulfate)175

Duponol OS (Oil soluble alkyl sulfate)176

Duponol WS (Oil soluble alkyl sulfate) 177

Egg yolk 178

Egg yolk powder and alkaline casein 179

Emulgor A 180

Emulphor AG oil soluble 181

Emulphor or methyl cellulose 182

Emulsifier L (Phosphate compound with wetting agent) 183

Emulsifier 157 184

Esters of abietic acid 185

Esters of montan wax 186

Ethanolamine 187

Ethanolamine sulfonates 188

Ethylene glycol monolaurate 189

Ethylene glycol monooleate 190

Ethylene glycol monostearate and stearate soap 191

Ethylene oxide, polymerized 192

Ethylene polymer with triethanolamine potassium oleate 193

Extract of oak bark 194

Fastolac (Mixture of vegetable gums) 195

Fatty acids having better emulsifying properties than neutral fats or synthetic fatty acid esters 196

Ferric oxide, colloidal 197

Filtra blend (Purified petroleum sulfonates) 198

Fish roe 199

Floex (Condensation product of higher fatty acids) 200

Fructose+, mannitol+ or sorbitol monostearate 201

Fuller's earth 202

Galactose 203

Galactose and pectin 204

Galagum C (Refined vegetable carbohydrate) 205

Gamboge 205a

Gardinol LS (Sodium oleyl sulfate)206

Gardinol WA (Sodium lauryl sulfate)207

Gelatin 208

Gelatin, deaminized 209

Gelatin and urea 210

GKS-5, a mixture of complex aromatic sulfonic acids and aromatic acids with aliphatic side chains <sup>211</sup>

Glucose monostearate 212

Glue with pyridinium octadecylbromide 213

Glue and starch 214

Glycerides, mono- or di-, of higher fatty acids 215

Glycerolated starch 216

Glycerol higher fatty acid esters polymerized, e.g., diglycerol stearate 217

Glycerophosphoric acid salts 218

Glyceryl monohydroxystearate 2180

Glyceryl monolaurate 219

Glyceryl monooleate 220

Glyceryl monoricinoleate S 221

Glyceryl monostearate and diethylaminoethyloleylamide 222

Glyceryl monostearate with diethylaminoethyl oleyl phosphate 223

Glyceryl monostearate and soap 224

Glyceryl oleate 225

Glyceryl oleyl triricinoleate 2258

Glyceryl (hexa, nona) ricinoleate 225b

Glyceryl tartrate 226

Glycol dioleate, polymerized 227

Glycol distearate, polymerized 228

Glycol glyceryl stearate or glycol glyceryl laurate 229

Glycol monolaurate, polymerized 280

Glycol monooleate, glyceryl dihydroxystearate, triethylene glycol monostearate <sup>231</sup>

Glycol monostearate, polymerized 232

Glycosterin (Diglycol stearate U. S. 1,914,100)238

Gomagel (Modified vegetable protein)284

Gum acacia 235

Gum dammar 286

Gum ghatti, sodium oleate, sodium glycocholate, albumin and lipose <sup>237</sup>

Gum tragacanth 238

Herrin milt 239

Higher fatty or chlorinated fatty acids with acid Turkey red oil or other sulfated oils <sup>240</sup>

Higher fatty acid ester of sugars or sugar alcohols 241

Humic acid or insoluble humic acid salts 242

Hydrohexalin (Hydralin) and soap 248

Hydromalin (Animated glyceride) 244

Hyglin (Water soluble cellulose ether)<sup>245</sup>

Idrapid (Octohydroanthracene mesasulfonic acids) 246

Igepal CA, CTA and CE (Polymerized ethylene oxide condensation product)<sup>247</sup>

Igepon A, Igepon T 248

Igepon AP extra (Oleic acid ester of aliphatic compound)<sup>249</sup> Igepon T <sup>250</sup>

Intramine (Sodium salt of sulfonated lauryl and myristyl collamide, U.S.P. 1,981,792)<sup>251</sup>

Iodine (Acts as an emulsifier for those water immiscible liquids in which its solutions exhibit particles under the ultramicroscope)<sup>252</sup>

Irish moss 253

Iron oleate 254

Iron tannate 255

Isopropyl valerate with gum arabic and tragacanth 256

Ivorit 257

Kelgin (Sodium alginate)<sup>258</sup>

Kleebatt emulsifier 259

Lamepon A (Condensation product protein derivative and fatty acids) 260

Lanette wax SX 261

Lanette wax SX, Lanette wax SXN (Mixture of fatty alcohols and emulsifying agent)<sup>262</sup>

Lanolin, solvent extract 263

Lead oleate 284

Lead salt of lauryl-1-sulfonate 265

Lead sulfate 266

Lecithin 267

Lecithin and benzyl alcohol 268

Lecithin and cholesterol 269

Lecithin and diglycerides of higher fatty acids 270

Lecithin hydrate 271

Lecithin and lactic acid 272

Lecithin, lysolecithin or lysocephalin 273

Lecithin, oxidized 274

Lecithin, oxidized soya bean 275

Lecithin and methyl cellulose 276

Lecithin and pine oil 277

Lexinol (Lecithin and associated phosphatides) 278

Lignin derivatives 279

Linseed aqueous extract 280

Lithium oleate 281

Lorol (Lauryl alcohol)<sup>282</sup>

Lorol (Technical dodecyl (Lauryl) alcohol)<sup>283</sup>

Lupogum (Locust bean gum)284

Magnesium chloride and cotton wadding 285

Magnesium oleate 286

Magnesium resinate 287

Mannide dilaurate 288

Mannide dipalmitate 289

Mannide monolaurate 290

Mannide monomyristate<sup>291</sup>

Mannide monooleate 292

Mannide monopalmitate 293

Mannide monoricinoleate 294

Mannide monostearate 295

Mannitan dilaurate 296

Mannitan laurate, modified 297

Mannitan monolaurate 298

Mannitan monolaurate, modified 298

Mannitan monomyristate 300

Mannitan monomyristate, modified 301

Mannitan monooleate 802

Mannitan monopalmitate 303

Mannitan monopalmitate, modified 304

Mannitan monoricinoleate 305

Mannitan monostearate 306

Mannitan monostearate, modified 307

Mannitan oleate 308

Mannitan tetralaurate 309

Mannitan trilaurate 310

Mannitol glyceryl hydroxystearate 310a

Mannitol glyceryl monolaurate 311

Mannitol glyceryl monooleate 312

Manucol V (Sodium alginate)<sup>313</sup>

Maprofix powder, Maprofix paste (Sodium borosulfate of technical cetyl to octadecenyl alcohol)<sup>314</sup>

Mapromol powder, Mapromol paste (Sodium borosulfate of technical octadecenyl alcohol)<sup>315</sup>

Melissyl calcium phosphate 316

Methocel (Methyl cellulose)<sup>817</sup>

Methyl cellulose 818

Methyl cellulose and cetyl alcohol 319

Methyl cellulose and lecithin 320

Miscibol 321

Modinal D paste (Long chain alcohol sulfate) 322

Modinal ES paste (Sodium sulfate of technical octadecenyl alcohol)<sup>323</sup>

Modinal ES paste (Sodium oleyl sulfate) 324

Monoaryl ethers of polyhydric alcohols with hydrotropic compounds of carboxylic or sulfonic acids of aliphatic, aromatic and hydroaromatic series 325

Monoethanolamine linoleate 326

Mono- or di- fatty acid glyceride and monofatty acid ester of glycols  $^{327}$ 

Monostearin 328

Monostearin sulfoacetate 329

Monostearyl esters of diethylene glycol, glycerol or diglycerol 330

Monostearyl glucose 331

Monostearyl glycerol, monostearyl diethylene glycol, dipalmityl glycerol <sup>332</sup>

Monostearyl mannitol 333

Morpholine oleate 334

Morpholine tetradecyl sulfate 335

Myristic esters of polyhydric alcohols 336

Nacconol NR (Sodium alkylaryl sulfonate)837

Naphtheic acids or acid naphthenic resin with caustic soda solution 338

Nekal A (Sodium octohydroanthracene mesasulfonate) 389

Nekal A E M 840

Nekal BX (Sodium naphthalene sulfonate)341

Nekal S 842

Nelgin (Modified colloidal silicate)343

Neomerpin 344

Neomerpin N 345

Nonaethylene glycol abietate, laurate, oleate or stearate 345a

Nopco 1216-K <sup>846</sup> Noremulsol <sup>847</sup>

Ocenol (Technical oleyl alcohol) 848

Octadecylamine soaps, adipyl, glycolyl or levulinyl 8480

Octylamine 349

Oil, Ahco base (Sulfonated compound) 350

Oil, base S-763A (Sulfonated mineral oil) 351

Oil or fat, phosphated or acetyl phosphated 352

Oil monopole (Double sulfonated castor oil) 853

Oil, oxidized coal tar oil 854

Oil, oxidized or polymerized vegetable and polyhydric alcohols partially esterified with higher fatty acids 355

Oil, oxidized soya bean 356

Oil, sunflower, oxidized 357

Oil, Turkey red 358

Oil, Turkey red, and diammonium hydrogenphosphate <sup>359</sup> Oleo glyceryl sulfate (Highly sulfated glyceryl fatty esters) <sup>360</sup>

Olevl or sulfonated fatty alcohols 361

Olibanum 361a

Oxycholesterol 362

Palmityl glycine 363

Palmityl and stearyl alcohols 364

Paremulsol 365

Pectin and fructose, sucrose and mannose 366

Pectin or hemicellulose 367

Pectin, milk, and sugar 368

Petronate (Sodium sulfated naphthenates) 369

Pharmagel (Specially refined gelatin) 370

Phosphated higher fatty alcohols 371

Phosphoproteins 372

Phosphoproteins, globulin, or albumin 373

Phosphoric acid esters of higher molecular weight 374

Phosphosol 375

Phosphotex 876

Phytosterols 377

Piperidine or organic acids 378

Pluratex (Alkylaryl sulfonate) 379

Polyglycerol fatty acid esters 380

Polyglycerol fatty ethers, e.g., diglycerol monolaurate 381

Polyhydric alcohol partially esterified with fatty acid amino products 382

Polyhydric alcohol partially esterified with higher fatty acid and an alkali \*\*8\*

Polyhydric alcohol partially esterified with a higher fatty acid and containing free fatty acid <sup>384</sup>

Polyhydric alcohol partially esterified with a higher fatty acid and sodium salt of a sulfated higher fatty alcohol <sup>385</sup>

Polymerized glycol higher fatty acid ester, e.g., polyglycol stearate 386

Polysulfonates of aromatic polyalkylene ethers 387

Polyvinyl alcohol 888

Polyvinyl alcohol 389

Polyvinyl alcohol sulfate 390

Potassium abietate 391

Potassium cetyl sulfoacetate 392

Potassium chaulmoograte 393

Potassium naphthenate 394

Potassium oleate 395

Potassium palmolate 396

Proflex 397

Proflex or hydromalin 398

Prolaurin (Propylene glycollaurate) 398

Propyl or butyl aromatic sulfonic acids 400

Propylene glycol monolaurate 401

Propylene glycol monooleate 402

Propylene glycol monoricinoleate 403

Propylene glycol monostearate 404

Protegin 405

Proteins, alkaline animal or fish 406

Proteins (hydrolyzed with alkali) 407

Protex gel (Aliphatic laurylamide sulfonated alkylbenzimidazol)408

Quaternary ammonium derivative of amino alcohol compound 409

Reaction product of an aminocarboxylic acid with an isocyclic carboxylic acid 410

Reaction product of polyvinyl alcohol and an aldehyde 411

Reaction product of a saponifiable wax or fat with a nitrogenous base 412

Reaction product of shellac with a nitrogen containing base, e.g., ammonia or methylamine 413

Reaction products of substituted thiocyanates and substituted amino acids 414

Red lead 415

Rosin or wax with polyglycerol monocarboxylic ester 416

Rosoap A 417
"Santomerse" (Alkyl aryl sulfonate) 418

Sapamine 419

Sapamines KW, MS (Salts of amides derived from higher fatty radicals)<sup>420</sup>

Sapogenin, betulin, ursolic, or oleanolic acid or their salts 421 Saponin 422

Saponin and alkali 428

Saponin and borax or ammonium carbonate 424

Saponin and methyl cellulose 425

Serum 426

Sherosope (Sodium sulfonaphthenate)427

Silica, clay or iron oxide 428

Soap, amine 429

Soap, n-butyl amine 480

Soap, castile 481

Soap, n-dibutylamine 482

Soap, 2-ethylhexylamine 488

Soap, guanidine 484

Soap, halogenated fatty acid 485

Soap, hydroxyalkylamine (Ethanolamine) 486

Soap, metallic 437

Soap, myristic acid 438

Soap, naphthenic 439

Soap and polyhydric alcohol partially esterified with higher fatty acid 440

Soap, potassium triethanolamine 441

Soap, sodium triethanolamine 442

Sodium abietate 443

Sodium alkyl naphthalene sulfonates 444

Sodium cerotate 445

Sodium cetyl phosphate 446

Sodium cetyl sulfonate 447

Sodium cholate 448

Sodium cholate 449

Sodium cholesteryl sulfoacetate 450

Sodium cymene sulfonate 451

Sodium elaidate 452

Sodium fatty alcohol sulfonate and G-cetyl betaine 458

Sodium glycocholate 454

Sodium glycol monooleate sulfate 455

Sodium heptadecyl sulfate 456

Sodium isopropylnaphthalene sulfonate and bone glue 457

Sodium lauryl sulfate 458

Sodium metasilicate 459

Sodium myristate or sodium palmitate 460

Sodium naphthenate 461

Sodium oleate 462

Sodium oleate, sodium myristate, sodium ricinoleate, sodium stearate, sodium resinate 463

Sodium petroleum sulfonate 484

Sodium polyacrylate 465

Sodium, potassium, ammonium or calcium gluconate 466

Sodium ricinoleate 467

Sodium salt of sulfonated stearic acid anilide 468

Sodium silicate 469

Sodium stearate 470

Sodium tetradecyl sulfate 471

Sorbide dilaurate 472

Sorbide monolaurate 473

Sorbide monomyristate 474

Sorbide monooleate 475

Sorbide monopalmitate 476

Sorbide monoricinoleate 477

Sorbide monostearate 478

Sorbitan dilaurate 479

Sorbitan monolaurate 480

Sorbitan monomyristate 481

Sorbitan monooleate 482

Sorbitan monopalmitate 483

Sorbitan monoricinoleate 484

Sorbitan monostearate 485

Sorbitan sulfonated ricinoleate 486

Sorbitan tetralaurate 487

Sorbitan tetrastearate 488

Sorbitan trilaurate 489

Sorbitol dilaurate 490

Sorbitol diolegte 491

Sorbitol distearate 492

Sorbitol glyceryl monolaurate 498

Sorbitol glyceryl monolaurate S284 494 Sorbitol glyceryl monolaurate S285 495

Sorbitol glyceryl monooleate S282 496

Sorbitol glyceryl monooleate S283 497 Sorbitol glyceryl monostearate 498

Sorbitol laurate 499

Sorbitol oleate 500

Sorbitol stearate A 501

Soya bean meal and caustic potash 502

Soya bean phosphatides 503

Soybean protein 504

Starch, dextrin, casein, Irish moss, sodium silicate or latex 505 Stearyl or cetyl alcohol and mixture of sodium salts of their

sulfonates 506

Stearyl glutamic acid 507

Stearyl sucrose 508

Stenol (Mixture of stearyl and cetyl alcohols) 509

Sterols 510

Strontium oleate 511

Sucrose 512

Sucrose, glucose or mannitol partially esterified with fatty acids 513

Sucrose oleate 514

Sugar house scum 515

Sulfanole C. P. (Alcohol fatty derivative) 516

Sulfanole P. B. (Aliphatic fatty derivative) 517

Sulfated fatty alcohol 518

Sulfated monoglycerides 519

Sulfated oleyl acetate 520

Sulfite 521

Sulfite cellulose lye 522

Sulfite liquor 523

Sulfite liquor, aliphatic or aromatic sulfonic acids 524

Sulfo dicarboxylic acids, e.g., octyl disulfosuccinic, etc.<sup>525</sup>

Sulfo fatty aromatic acids 526

Sulfonates, alkali, high molecular, e.g., sodium tetrahydronaphthalene- $\beta$ -sulfonate  $^{527}$ 

Sulfonate, petroleum 528

Sulfonated cocoa butter or stearin 529

Sulfonated distillation product of glycol and olive oil 530

Sulfonated fatty acids 581

Sulfonated fatty hydroxy acid esters 532

Sulfonated higher fatty acids 533

Sulfonated hydrogenated castor oil 534

Sulfonated hydrogenated castor oil, ammonium salt 535

Sulfonated hydrogenated castor oil, isopropylamine 536

Sulfonated hydrogenated castor oil, potassium salt 587

Sulfonated hydrogenated castor oil, sodium salt 588

Sulfonated hydrogenated caster oil, triethanolamine 539

Sulfonated oil, castor 540

Sulfonated oil, coconut 541

Sulforated oil, fish, egg, apricot, peach-kernel, almond or teaseed  $^{542}$ 

Sulfonated oil, mineral and benzol 543

Sulfonated oil, mineral, condensed with alcohols 544

Sulfonated oils, oxidized, e.g., oxysulfoleic acid 545

Sulfonated oil, palm 546

Sulfonated oil, red 547

Sulfonated oil, Turkey red 548

Sulfonated oleylbutyrate or other sulfonated higher fatty alcohol esters 549

Sulfonated polymerized oils, fats or fatty acids 550

Sulfonated propylene glycol ricinoleate 551

Sulfonated spermaceti, beeswax or woolfat 552

Sulfonated squalene 553

Sulfonated stearic or palmitic acids 554

Sulfonated stearin pitch 555

Sulfonated stearin pitch 556

Sulfonated tallow 557

Sulfonated wool fat fatty acids 558

Sulfonic acid, aromatic, with at least one alkyl group and a light metal salt 559

Sulfonic acid, substituted aromatic, e.g., amyl naphthalene- $\beta$ -sulfonic acid  $^{660}$ 

Sulfonic acid, unsaturated 561

Sulfuric esters of amides or anilides 562

Sulfuric ester of higher fatty alcohol 568

Tegacid 564

Tegin (glyceryl monostearate) 565

Tegin P (Propylene glycol monostearate) 566

"Tergitol" penetrant (Sodium sulphate of higher alcohol) 567 Tetraethylene glycol stearate 567a

Tetrahydronaphthalene sulfonic salts 568

Tetrasodium pyrophosphate (Anhydrous powder & flakes) 569 Thoreps 570

Triethanolamine 571

Triethanolamine and other alkylolamines 572

Triethanolamine condensed with stearic sulfonic or sulfonated carboxylic acids 573

Triethanolamine naphthenate 574

Triethanolamine (mono, di or tri) laurates 574a

Triethanolamine stearate 575

Triethanolamine tetradecyl sulfate 576

Trigamine 577

Trigamine stearate 578

Triglyceride, partially saponified, containing soap, e.g., glyceryl mono- and distearate 579

Trihydroxyethylamine stearate 580

Trihydroxyethylamine stearate, special 581

Trimethylamine 582

Trimethylol aminomethane 583

Triton NE (High molecular weight complex alcohol) 584

Triton W-30 (Sulfated aromatic ether alcohol) 585

Triton K-60 (Cation active quaternary salt) 586

Triton 720 (Sulfonated ether) 587

Triton E-79 (Amine salt of a sulfated ether) 588

Turkelene 589

Turpentine, spruce 590

Tylose (Methyl cellulose) 591

Urethane oleate 592

Urethane oleate (Cyclic) 593

Ursolic acid 594

Vaneps 595

Warcosol (Alkyl naphthalene sulfonate) 596

Wax, saponifiable, e.g., montan, I. G., carnauba, Rilan; with borax or ammonium bicarbonate 597

Whey protein 598

Wool fat and phenol, sulfonated 599

Xerol (Glyceryl monostearate) 600

Xynomine powder, Xtnomine paste (Sulfated boroamide ester of aliphatic compounds containing 12 to 18 carbon atoms, U. S. 1,918,373) 601

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## CHAPTER VIII

## LIST OF EMULSIONS

Acriflavine, petrolatum and mineral oil, with wool fat 1

Acrylic nitrile and butadiene, with diethylamine-ethoxyoleylanilide hydrochloride <sup>2</sup>

Acrylic nitrile and butadiene, with sodium oleate 3

Alcohol, with potassium-cupric tartrate 4

Aliphatic or hydroaromatic alcohols, ketones, hydrocarbons, higher oleic acid or other fatty substances, aromatic bases or dyes with sodium dicresyl phosphate <sup>5</sup>

Amide, octadecyl, myristyl, or lauryl, with stearic acid and sodium silicate  $^6$ 

Amides, higher fatty, (e.g., stearamide), with sodium silicate and a higher fatty acid  $^{7}$ 

Amyl acetate and boric acid, with "Emulgor-A" 8

Amyl alcohol, amyl acetate or ethylacetate, with iodine 9, 10

Aniline or dimethylaniline, with soap 11

Aromatic principles, with ammonium sulforicinoleate 12

Asphalt, with alkali and alkaline casein 13

Asphalt, with alkali and clay 14

Asphalt, with alkali phosphatides 15

Asphalt with alkali and soap or clay 16

Asphalt, with alkali tannate 17

Asphalt, with aluminum hydroxide and asbestine 18

Asphalt, with ammonia and casein 19

Asphalt, with bentonite 20

Asphalt, with colloidal clay and anhydrous soap and olein <sup>2</sup> Asphalt, with colloidal hydrous magnesium silicate <sup>22</sup>

Asphalt, with fatty acid and sodium or potassium carbonate <sup>28</sup>
Asphalt, with ferrous sulfate, potassium tannate and potassium caseinate <sup>24</sup>

Asphalt, with flaxseed mucilage 25

Asphalt, with gelatin or glue 26

Asphalt, with gelatin and salts 27

Asphalt, with glue and soap 28

Asphalt, with green petroleum acid soap and corn gluten, or soya-bean meal <sup>29</sup>

Asphalt, with "Kontakt" or naphthenic acid 80

Asphalt, with montan wax and caustic soda 31

Asphalt, with naphthenic soap 32

Asphalt, with oleic acid and borax or sodium silicate 33

Asphalt, with oxidized paraffin wax soap 34

Asphalt, with potassium tannate or caseinate 35

Asphalt, with silica gel 36

Asphalt, with soap and bentonite 37

Asphalt, with soap or clay 88

Asphalt, with soap and colloidal clay 89

Asphalt, with soap, electrolytes or colloids 40

Asphalt, with sodium abietate 41

Asphalt, with sodium cresylate 42

Asphalt, with sodium fatty alcohol sulfonates 43

Asphalt, with sodium petroleum sulfonate 44

Asphalt, with sodium phosphate 45. 46

Asphalt, with sodium or potassium peroxide or percarbonate 47

Asphalt, with sodium silicate and oleic acid 48

Asphalt, with soya bean flour 49

Asphalt, with starch, borax 50

Asphalt, with starch or dextrin and alkali 51

Asphalt, with stearin pitch and caustic soda 52

Asphalt, with sulfate lignin and metallic salts 58

Asphalt, with tannic acid or synthetic tannin 54

Asphalt, with triethanolamine oleate and a salt 55

Asphalt, with trisodium phosphate 56, 57, 58

Asphalt, with wax acid metallic soap and caustic soda 50

Asphalt, bitumen, coal-tar pitch, cresol or dead oil, with loam, soil or clay 60

Asphalt and carbon tetrachloride, with colloidal clay 61

Asphalt, cumarone resin and montan wax, with sodium or potassium hydroxides and methyl cellulose 62

Asphalt and fatty acid, with alkali silicate or alkali borate 63 Asphalt, fatty acids, oil or tar, with humic acids or humates 64

Asphalt and fuel oil, with soap and metallic soap 65

Asphalt and gasoline, with soap 66

Asphalt, glycerides and mineral oil, with potassium soap 67 Asphalt and montan wax, with alkali 68

Asphalt or other bituminous material, with protein (casein), starch or oleic acid and alkali 69

Asphalt or pitch, with bentonite and acid 70

Asphalt or pitch, with naphthenic or fatty acid scap 71

Asphalt or pitch, with petroleum and alkali 72

Asphalt, pitch, and petroleum, with lead oxide 73

Asphalt or pitch, with sulfonated fatty oils 74

Asphalt or pitch, with sulfonated mineral oil 75

Asphalt, pitch or rubber, with viscose 76

Asphalt, pitch or tar, with colloidal clay and magnesium-oxide or hydroxide 77

Asphalt residue or bitumen, with soap, rosin soap or rosin oil soap 78, 79

Asphalt, resins, mineral oil, tar, vegetable or animal oils, with sulfonated fatty acids  $^{80}$ 

Asphalt and rosin oil, with sodium abietate 81

Asphalt, rosin and petroleum pitch, with Turkey red oil 82

Asphalt or tar, with cholesterol or soap 83

Asphalt or tar, with lecithin or other phosphatides 84

Asphalt, tar or pitch, with oil cake and alkali 85

Asphalt, wax tailings and rosin, with colloidal clay 86

Bakelite C-9 resins, with casein and ammonia and morpholine 87

, Balsam Peru with soap bark and saponin 88

Beeswax, with potassium carbonate 89

Beeswax and p-cymene, with ethyl cellulose 90

Bees, Japan, or montan wax, with potassium carbonate 91

Beeswax, stearine and ether, with ammonia 92

Benzene with agar, saponin, albumin, pectin, gelatin, lecithin or casein 93

Benzene, with castor oil soap, sodium oleate or coconut oil soap or kaolin 94

Benzene, with colloidal ferric hydroxide 95

Benzene, with methanol, ethanol, allyl alcohol, phenol, resorcinol or higher fatty acids <sup>96</sup>

Benzene, with phenol or resorcinol 97

Benzene, gasoline, naphtha, or kerosene, with trihydroxyethylamine stearate 98

Benzene and latex, with soap 99

Benzene, nitrobenzene, chloroform or carbon tetrachloride and cottonseed oil, with gelatin 100

Benzene, toluene, xylene, aniline, o-toluidine, nitrobenzene or dimethylaniline, with soap 101

Benzine, with saponin 102

Benzine and beeswax, with caustic soda and soap 103

Benzine, benzene, petroleum, toluene, xylene, tetralin, decalin, hexalin, methyl hexalin, carbon tetrachloride or trichlorethylene, with Turkey red oil, Monopole oil, ammonium oleate, triethanolamine, Lanette Wax SX, or Betan (sodium  $\beta$ -tetralin sulfonate)<sup>104</sup>

Benzine and benzol, with soap 105

Benzine or mineral oil, with "Savonade" (methyl hexalin and potassium oleate) 106

Benzine or trichlorethylene with acidified carrageen, agar or lrish moss 107

Benzoic-phthalic glyceride, with ammonia and gum arabic or gum tragacanth or Irish moss 108

Bitumen, with acid sulfonated oil and alkali 109

Bitumen, with alkali hydrolyzed glyceride 110

Bitumen, with alkaline sulfonated oil 111

Bitumen, with alkali salts of  $\alpha$  or  $\beta$  naphthols, pyrogallol, resorcinol or carbazol 112

Bitumen, with alkali tannate 113

Bitumen, with bentonite 114

Bitumen, with bentonite and calcium caseinate 115

Bitumen, with blood 116

Bitumen, with borax 117

Bitumen, with calcium oxide and alum 118

Bitumen, with calcium resinate and sodium resinate 149

Bitumen (asphalt), with casein and alkali 120

Bitumen, with chlorinated tall oil 121

Bitumen, with formaldehyde phospho-protein condensation 122

Bitumen, with humic acid and salt 123

Bitumen, with humic or lignic acid 124, 125

Bitumen, with hydrochloric acid and caustic soda 126

Bitumen, with iron, aluminum or chromium sulfate and waste sulfate liquor 127

Bitumen, with iron hydroxide, aluminum hydroxide, xinc hydroxide, basic magnesium salts or alkaline starch 128

Bitumen, with lignic acid or oxidized tar extract 129

Bitumen, with naphthenic or rosin soap and casein 130

Bitumen, with protein or starch 131

Bitumen, with rosin and alkali 132

Bitumen, with rosin soap and milk 133

Bitumen, with rosin soap and molasses 134

Bitumen, with rubber latex 185

Bitumen, with soap and alkaline casein and anthracene oil or creosote 136

Bitumen, with soap and calcium resinate 187

Bitumen, with soap and casein 138, 139

Bitumen, with soap and gelatin 140

Bitumen, with sodium hydroxide and sodium phenate 141

Bitumen, with sodium oleate 142

Bitumen, with sulfite liquor concentrate 143

Bitumen, with sulfurized oils or sulfurized fatty acids 144

Bitumen, with tall oil soap 145

Bitumen, with water or alkali soluble phenol formal dehydecondensation  $^{146}\,$ 

Bitumen, with water swelling gum 147

Bitumen or asphalt, with latex 148

Bitumen or asphalt, with starch and alkali 149

Bitumen, asphalt, petroleum or coal tar, with soap or rosin soap 150

Bitumen and benzine, with barium chromate suspended in hot water 151

Bitumen and gasoline, with potassium dichromate 152

Bitumen or oil, with rubber latex 153

Bitumen or pitch, with clay 154

Bitumen, resin or wax, with sodium silicate 155

Bitumen, resin and wax, with sulfite cellulose extract 156

Bitumen, tar, creosote, or petroleum, with alkaline casein or alkaline blood albumin 157

Bitumen, tar or pitch, with casein, rosin and caustic soda 158 Bitumen and turpentine, with sodium hydroxide 159

Bitumen and Vinsol resin, with alkali 160

Borneol, terpene oil, fenchyl alcohol, with soft soap 161

Butadiene, with diethylaminoethyloleylamide hydrochloride 162

Butadiene, with (substituted) oleyl alcohol 163

Butadiene, with saponin 164

Butadiene, with sodium isoproplynaphthalenesulfonate 165

Butadiene, with sodium and magnesium oleates 166

Butadiene, with sodium oleate and magnesium oleate 167

Butadiene, with sodium oleate, sodium isobutyl naphthalene sulfonate or albumin or diethylaminoethyloleylamide or 2-npentadecylglyoxalidine; sodium oleate 168, 169

Butadiene, with sodium stearate 170

Butadiene and butylene, with ammonia soap, glucosides or alginic acid 171

Butadiene or isoprene, with salts of sulfonic or carboxylic acids or their amides 172

Butadiene or isoprene, with sodium stearate 173

Butadiene or styrene, with sodium stearate 174

Butadiene and vinylidene chloride, with sodium oleate 175

Butter, with egg white, gelatin, agar or gum arabic 176

Butter, with lecithin 177

Butter, with skim milk 178, 179

Calcium sulforicinoleate, with gelatin or casein 180

Camphor and acetaldehyde, with sodium oleate 181

Carbon bisulfide, with alcohol and potassium oleate 182

Carbon bisulfide, with potassium rosin fish oil soap or sodium rosin oleic acid soap 188

Carbon bisulfide, with sulfonated castor oil 184

Carbon bisulfide and butyl or amyl alcohol, with soap 185

Carbon bisulfide or oils, with ammonia or amine and zinc. copper, or nickel hydroxide 186

Carbon bisulfide and olein, with potash soap 187

Carbon bisulfide and sulfur, with fish oil soap 188

Carbon tetrachloride, with decylcarboxymethylisothiourea hydrobromide. 189

Carbon tetrachloride with triethylammoniumoleate 190

Carbon tetrachloride, aniline, n-octanol, ethyl oleate, chloroform, benzene, toluene, xylene, higher paraffins, carbon bisulfide or hexane, with sodium oleate 191

Carbon tetrachloride and benzene, with sodium oleate 192

Carbon tetrachloride, benzene or camphor, with wool fat or sulfonated wool fat fractions 193

Carbon tetrachloride, chloroform, benzene, turpentine, almond or mineral oil, with gum tragacanth, gum acacia or Irish moss 194

Carbon tetrachloride and cyclohexanol, with ammonium oleate 195

Carbon tetrachloride and light oils, with saponin, so ap or gum arabic  $^{196}$ 

Carbon tetrachloride, tetrachlorethane, trichlorethylene, tetrahydronaphthalene, benzene or naphtha, with sodium or ammonium sulforicinoleates <sup>197</sup>

Carnuaba wax (bright drying), with alkanolamine soap 198

Carnauba wax, with ammonia soap or glyceryl monostearate 199

Carnauba wax, with morpholine oleate 200

Carnauba wax, with soaps of monoethanolamine, diethanolamine, triethanolamine or morpholine 201

Carnauba wax, with sodium and potassium stearates 202

Carnauba wax, with trihydroxyethylamine-linoleate or stearate  $^{208}$ 

Carnauba wax and carbon tetrachloride, with lanolin and gelatin 204

Carnauba wax and kerosene, with ammonium linoleate 205

Carnauba and paraffin waxes, with starch 206

Carnauba wax and resin, with soaps 207

Carnauba wax and shellac, with borax and soap 208

Castor, apricot or almond oils, with starch 209

Cellulose nitrate and dibutyl phthalate, with gelatin 210

Cetyl alcohol and shellac, with ammonia and triethanolamine 211

2-chlorbutadiene, with diisobutyl-naphthalene sulfonate and sodium oleate <sup>212, 213</sup>

Chlorobutadiene, with sodium oleate, quaternary ammonium salts or sodium fatty alcohol sulfonates <sup>214</sup>

Chlorobutadiene, with sodium oleate or sulfonated castor oil 218

Chloroform, with silver dichromate 216

Chloroprene polymers, with C-cetyl betaine 217

Chloroprene or styrene, with partially saponified polyvinyl acetate or partially saponified formaldehyde polyvinyl acetal <sup>218</sup>

Chlorothymol, with alkaline casein 219

Chlorothymol, with rosin soap 220

Chocolate, with lecithin 221

Cocoa butter, with gelatin and methyl cellulose 222

Cocoa butter, with lecithin, cholesterol or cetyl alcohol 223

Copper oleate and butanol, with sodium acetate 224

Copper, zinc, silver, calcium, manganese or cobalt resinates, with ammonia and casein, soy protein or gelatin <sup>225</sup>

Cresol, with linseed oil soap 226

Cresol, with sodium stearate 227

Cresylic acid, with soap 228

Cresylic acid, phenol, o-cresol, m-cresol, p-cresol, with potassium oleate <sup>229</sup>

Cyclohexanol, with soap 280

Cyclohexanol or methyl cyclohexanol and tetralin or decalin, with lecithin <sup>231</sup>

Dibenzanthracene, benzanthracene, dihydroanthracene or chrysene, with gelatin <sup>232</sup>

Dibenzanthracene and pyridin, with gum acacia 233

'Dibutyl phthalate, with bentonite and soap 284

Dichlorbenzene, cresol and camphor oil, with sulfonated oils 235 Diethylene glycol abietate, with "Duponal ME," sulfated castor oil and soap 236

Diethyleneglycol abietate, "Abalyn," or "Hercolyn," with alkaline casein with or without sulfonated oil and Dupanol 237

Diethylphthalate, with soap 238

Dimethylerythrene, with soap 239

Dimethyl sulfide, with calcium humate 240

Diolefins, with milk, glue or gelatin 241

Diolefins (e.g., butadiene), with proteids, milk, dextrin, soap or sulfonates 242

Diolefins, with soap or sulfonic acid salts 243

Dipentene, with sodium butylnaphthalene sulfonate or sodium isopropylnaphthalene sulfonate 244

Dyes, with bentonite and casein 245

Egg yolk substitute, with lecithin and improved with hydrogen peroxide  $^{246.247}$ 

Erythrene, isoprene or butadiene and styrene, with sodium stearate 248

Ethanol-stearamide, with sodium silicate and higher-fatty acid 249

Ether, ethyl acetate, amyl acetate and amyl alcohol, with iodine  $^{250}$ 

Ethylenedichloride, with magnesium hydroxide, starch, gelatin, glue or albumin  $^{251}$ 

Ethylenedichloride, with potash fish-oil soap <sup>252</sup> Ethylenedichloride, with sulfonated sperm oil <sup>253</sup>

Ethylenedichloride and oleic acid, with diglycol oleate and ammonia 254

Ethylenedichloride or trichlorethylene, with Turkey red oil and bentonite 255

Fat, with egg yolk 256

Fat, with gelatin 257

Fat, with malt or malt extract 258

Fat, with powdered gum arabic or tragacanth 259

Fat, animal, with lecithin and skim milk 260

Fat, edible, with skimmed milk, lecithin and malt extract <sup>261</sup> Fats, butter and vegetable, with skim milk <sup>262</sup>

Flax wax, with sodium oleate or ethanolamine 263

Furfural, engine-, paraffin oil or kerosene, with bentonite <sup>264</sup> Gasoline, with alginic acid, casein and sodium silicate <sup>265</sup>

Gasoline, with copper oleate and ammonium nitrate 266

Gasoline, with petroleum sulfonic acid 267

Gasoline, with soap 268. 269

Gasoline, with sodium, ammonium or triethylammonium oleate  $^{270}$ 

Gasoline, with triethylammonium oleate 270

Gasoline, ether, benzene, or chloroform, with wool fat fractions or their sulfonates <sup>271</sup>

Gasoline and eucalyptus oil, with soap 272

Gilsonite, with tannic acid 273

Gilsonite and hydrocarbon solvent, with glue 274

Glyceryl phthalate (in solvent) with bentonite, soap or gelatin 275

Glycol abietate, xylene and butanol, with sulfonated tallow and caustic soda 276

Gum benzoin, with gum tragacanth 277

Gum chicle, with bentonite, casein, sodium stearate, albumin, starch, flour or soap <sup>278</sup>

Gum, resin, rubber or wax and solvent with soap or sulfonated oil <sup>279</sup>

Gum sandarac, with ammonium oleate 279

Gurjun balsam, with neutral sulfonated castor oil 280

Heptane and carbon tetrachloride, with kaolin 281

Hydrocarbons, with hydrocarbon sulfones and sulfonic acids 282

Hydrocarbons, with naphthenic acids or naphthene sulfonic acids <sup>283</sup>

Hydrocarbons, oils or waxes, with one from each of two following groups: (A) protein, agar, saponin or bile; (B) starch, dextrin, rubber or vegetable glue 284

Hydrocarbons or substituted hydrocarbons, e.g., trichlorethylene, with soap and silica gel <sup>285</sup>

Hydroxybiphenyl with Turkey red oil and diphenyl ether or guaiacol <sup>286</sup>

Isobornyl acetate, with methyl cellulose 287

Isobutyl p-aminobenzoate (cycloform), with lanolin 288

Isoprene, with ammonium oleate and casein 289

Isoprene, with sodium or ammonium oleate or Turkey red oil 290

Isoprene or butadiene, with albumin or soap or sodium isobutylnaphthalene sulfonate <sup>291</sup>

Isoprene and dibutyl fumarate, with Turkey red oil and sodium alkyl naphthalene sulfonate <sup>292</sup>

Isoprene and dimethylbutadiene, with albumin, gelatin or hemoglobin <sup>293</sup>

Isoprene or dimethyl butadiene, with ammonium or potassium oleate and casein or albumin <sup>294</sup>

Isoprene or erythrene, with diethylaminoethyloleylamide hydrochloride or diethylaminoethoxyoleylanide hydrochloride <sup>295</sup>

Japan wax, with soap 296

Kerosene, with ammonium oleate 297, 298

Kerosene (SO<sub>2</sub> extract), with arsenic compounds 299

Kerosene, with butyl acetyl ricinoleate 800, 801

Kerosene, with carbon black 302, 303, 304

Kerosene with soap 305

Kerosene, with sodium oleate, potassium stearate or potassium balmitate 306

Kerosene, with sodium stearate 807

Kerosene, and alkyl thiocyanates with triethanolamine oleate 308 Kerosene or benzene, with arsenious sulfide or antimony trisulfide or silica 309

Kerosene, and dibutyl phthalate, with soap 810

Kerosene and linseed, with soap and starch 311

Kerosene, olive or castor oil, with sodium oleate 312

Kerosene or pine oil, benzine, toluene, or chlorinated solvents, with Dupanols 313

Kerosene, pyrethrum and methyl salicylate, with soap 314

Lacquer, with gum ghatti and sodium isopropylnaphthalene sulfonate  $^{315}$ 

Lacquer, with reaction product of ethylene oxide and castor oil 816

Lanolin, with calcium icthosulfonate 317

Lard, butter, or olive oil, with egg yolk or albumin 318

Lard and hydrogenated cotton seed oil, with acid sodium stearate 319

Lard or mineral oil, with soap 320

Lard and sesame oil, with caustic potash 821

Lead arsenate, with oleic acid and triethanolamine 322

Lead arsenate, soya bean oil, with lime 828

Lethane, with sulfite lye 324

Methylbutadiene, with soap 325

Methylmethacrylate, monomeric, with Turkey red oil and sodium diisopropyl naphthalene sulfonate 326

Montan wax, with magnesium sulfate and sodium silicate 827 Naphthalene, chlorinated, with hydrogenated naphthalene 328

Naphthalene, chlorinated, and decahydronaphthalene with sulfite-cellulose extract 323

Naphthalene, hydrogenated, with sulfonated fat or sodium alkylated aromatic sulfonate  $^{380}$ 

Nicotine, with soap 331

Nicotine, derris or pyrethrum extracts, with sulfonated petroleum 332

Nitrobenzene, with polyiodides and electric current 833

Nitrocellulose, with pectin or agar 334

Nitrocellulose, with soap, sulfated terpene, or higher alcohol, gelatin, Igepon or sulfonated castor oil <sup>835</sup>

Nitrocellulose, with sodium resinate or sodium lauryl sulfate 336

Nitrocellulose and cyclohexanone, with sodium oleate 337

Nitrocellulose lacquer, with gelatin or sodium oleate 338

Nitrocellulose, resin and solvent, with soap gelatin or alkaline casein 339

Nitrocellulose and solvents, with alizarin oil 340

Nitrocellulose, solvents and oils, with magnesium, calcium or barium soaps and resins, e.g., dammar <sup>341</sup>

Nitrocellulose and wood creosote, with gum tragacanth 342

Octane, with sodium oleate 343

Oil, with ammonia soap 344

Oil, with bentonite and soap 345

Oil, with bentonite and vegetable gum 346

Oil, with blood-albumen and ammonia 847

Oil, with cetyl or stearyl alcohol and sulfated higher fatty alcohol sodium salt 348

Oil, with lecithin 849

Oil, with lecithin and hydrogen peroxide 850

Oil, with silica, clay or iron oxide 351

Oil, almond, with gum arabic and magnesium carbonate 352

Oil, almond, and beeswax, with a higher aliphatic acid ester of an aliphatic polyhydroxy substance and an aliphatic polycar-boxylic acid 358

Oil, anthracene, with sulfite cellulose extract 855

Oil, anthracene, or wood; or chlorine compounds, with bentonite 356

Oil and bitumen, with acetylene 357

Oil, blubber, with soda ash 858

Oil, camphor with soap 359

Oil, castor, with agar 360

Oil, castor (blown), with ammonium ricinoleate 361

Oil, castor, with gelatine 362

Oil, castor, with gum acacia 363

Oil, castor, with linseed and castor oil soap 364

Oil, castor, with sodium hydroxide, or milk magnesia; gelatin and sugar 365, 366

Oil, castor, with sodium hydroxide or potassium hydroxide 367

Oil, castor, with tannin 368

Oil, castor, almond, cod liver, cottonseed, linseed, olive, or sesame, with gum acacia  $^{369}$ 

Oil, castor, carbon disulfide and alcohol, with potassium hydroxide 370

Oil, hydrogenated castor, with triethanolamine stearate 371

Oil, castor, linseed, cod liver or cottonseed, with gum arabic 372

Oil, castor (blown), and mineral oil, with ammonia 373

Oil, castor and mineral, with pectin 374

Oil, castor or olive, with morpholine 375

Oil, castor, olive or cod liver, with soap or calcium or barium hydroxide 876

Oil, castor, rapeseed, and turpentine, with ammonia 377

Oil, coal tar, with sulfonated castor oil 378

Oil, coal-tar, creosote, tar, petroleum, pitch, bitumen or asphalt, with casein, rosin and caustic soda or potash 379

Oil, coconut, with casein and ammonia 380

Oil, coconut, with flour or starch 381

Oil, coconut, with gelatin 382

Oil, coconut, with (skim) milk 383, 384

Oil, coconut, with soap 385

Oil, coconut, with soya bean phosphatide 386

Oil, coconut, cottonseed or peanut, or butter, with cetyl alcohol and milk 387

Oil, coconut or peanut, with polyglycerol ester, lecithin or glyceryl laurate 388

Oil, coconut, and stearin, with milk 389

Oil, coconut and "Vaseline," with caustic soda 300

Oil, cod liver, with alkali metaphosphate 391

Oil, cod liver, with calcium phosphorus organic complex and gum acacia 392

Oil, cod liver, with gum arabic 393

Oil, cod liver, with methylcellulose 394

Oil, cod liver, with pectin 395

Oil, cod liver, with protalbic acid 396

Oil, cod liver, with protalbic acid, casein, calcium, saccharate egg yolk and lime water 396, 397, 398, 399

Oil, cod liver, with soap, gum arabic and gelatin 400

Oil, cod liver, with sodium metaphosphate, tragacanth and gum arabic 401

Oil, cod liver, with sodium silicate and aluminum sulfate402

Oil, cod liver, or olive, with alkali casein 403

Oil, cottonseed, with alkali soaps 404

Oil, cottonseed, with gum acacia 405

Oil, cottonseed (hydrogenated), with lecithin and triethanolamine 408

Oil, cottonseed, with milk or gum arabic 407

Oil, cottonseed, with pectin 408

Oil, cottonseed and calcium carbonate, with "Emulgor A" and oleic acid 409

Oil, cottonseed or corn, with sodium stearate or ammonium stearate 410

Oil, cottonseed, corn, linseed, olive, rapeseed, peanut, sesame, castor or sperm, with soap 411

Oil, cottonseed or mineral, or turpentine or benzene or carbon tetrachloride or chloroform or hexane, with gum acacia or soap 412

Oil, cottonseed or other vegetable oil, with sulfonated coconut and castor oils 413

Oil, cottonseed or red engine, with p-dichlorbenzol and sodium bicarbonate 414

Oil, cottonseed, and tallow, with soap 415

Oil, cranberry seed, with ursolic acid 416

Oil, creosote, with gelatin 417

Oil, creosote, with Nekal A.E.M. (glue and sodium isopropylnaphthalene sulfonate)<sup>418</sup>

Oil, creosote, with rosin soap 419

Oil, creosote, with sodium hydroxide 420

Oil, diesel, with bentonite 421

Oil, dill weed, with gum tragacanth 422

Qil, drying (e.g., linseed oil), with alkaline casein 423

Oil, drying, with glue 424

Oil, drying, with sodium hexametaphosphate 425

Oil, drying, with triethanolamine soaps 426

Oil, drying, and resins, or varnish, with sodium alginate 427

Oil, egg yolk, with lecithin and cholesterol 428

Oil, essential, with albumin and potassium bitartrate 429

Oil, essential, with potassium oleate, triethanolamine linoleate or ammonium ricinoleate sulfate \*30

Oil, essential, with skim milk 481

Oil, essential, with sugar solutions 432

Oil, essential, with sulfonated fatty oil 488

Oil, essential or etherial, or salol, with Turkey red oil sulfonaphthenic acids or purified sulfite cellulose liquor 484

Oil, essential, and stearopten, with soft soap 485

Oil, eucalyptus, with rosin soap 436

Oil and fat, with albumin 437

Oil or fat, with aluminum hydroxide and urea 438

Oil or fat, with esters of sulfo-fatty acids and albumin or glue 489

Oil or fat, with glycerin and starch 440

Oil or fat and solvent and radioactive substance, with caustic soda 441

Oil, fatty, with chlorhydrin or diethylene dioxide 442

Oil or fatty acid, with soyabean phosphatides or lecithin 443

Oil, fish, with casein and disodium phosphate 444

Oil, fish, asphalt, petroleum, coal tar pitch, or cresol, with sulfite cellulose lye and colloidal silica 445

Oil, fish or whale, with rosin soap 448

Oil, flavoring, with gum acacia 447

Oil, fuel, with clay or soap 448

Oil, fuel, with starch or casein emulsion 449

Oil, fuel, or coal, with nitrogen monoxide and casein or starch 449, 450

Oil, fuel or kerosene, with alkali-or triethanolamine soap 451

Oil, gas, with naphthenic soap 452

Oil and glycol or glycol ether, with sulfonated oil 458

Oil, hop extract, with malt syrup 454

Oil, hydrogenated vegetable, with polymerized and oxidized glycerides 455

Oil, linseed, with animal blood 456

Oil, linseed, with antimony chloride 457

Oil, linseed (boiled), with Duponol WA and caustic potash 458

Oil, linseed, with egg albumin 459

Oil, linseed, with glue and soda ash 460

Oil, linseed, with potassium linoleate 461

Oil, linseed, with sodium resinate and ammonium sulfate 462

Oil, linseed, with sugar and starch 463

Oil, linseed, butadiene and oleic acid, with Turkey red oil 464

Oil, linseed, castor or soya bean, with ethanolamine oleate or hydroxyethylamine naphthenate 465

Oil, linseed or castor, turpentine and cellulose acetate, with gelatin, glue and casein solution 486

Oils, linseed and China wood, with casein, glue and flour 437

Oil, linseed, China wood and gilsonite, with rosin soap 468

Oil, linseed, and p-chlormetacresol, with potassium soap 469

Oil, linseed, ester gum, and rosin, with triethanolamine 470

Oils, linseed and mineral, with antimony chloride 471

Oil, linseed, and oleic acid, with china clay, fuller's earth or bentonite 472

Oil, linseed, poppy or soya bean with soap, saponin, sulforicinates, carragheen moss or gelatin 473

Oil, linseed, "Pyroxylin," butyl acetate and gasoline, with sodium oleate 474

Oil, linseed, and rosin, with glue 475

Oil, linseed, tallow and rosin, with sodium hydroxide 476

Oil, lubricating (no added emulsifier)477

Oil, lubricating, with bentonite 478

Oil, lubricating, with calcium oxide 479

Oil, lubricating, with electric discharge 480

Oil, lubricating, with kaolin, glue or skim-milk 481

Oils, lubricating and pine, with sodium naphthenate 482

Oil, marine, with disodium hydrogen phosphate 483

Oil, mineral, with agar 484

Oil, mineral, with agar and acid 485

Oil, mineral, with agar; malt extract; casein or egg yolk; psyllium-seed, agar, and gelatin 486, 487, 488, 489, 490

Oil, mineral, with alkali metal naphthenate and monethyl ether of ethylene glycol <sup>191</sup>

Oil, mineral, with alkali soap of sulfonated petreleo and glue  $^{492}$ 

Oil, mineral, with aluminum naphthenate and partially esterified glyceryl oleate  $^{493}$ 

Oil, mineral, with ammonium laurate 494

Oil, mineral, with barium hydroxide 495

Oil. mineral, with barium soap 496

Oil, mineral, with basic copper sulfate, basic ferrous sulfate, basic nickel sulfate, ferrous hydroxide or ferric hydroxide 497

Oil, mineral, with bentonite 498, 499

Oil, mineral, with calcium stearate 500

Oil, mineral, with cyclohexylamine oleate 501

Oil, mineral, with diglycol laurate, diglycol oleate or sodium olevl sulfate  $^{502}\,$ 

Oil, mineral, with dioctade cenyl ether of trioxytriethylamine-acetate  $^{503}$ 

Oil, mineral, with Fuller's earth 504

Oil, mineral, with glue and sodium oil soluble mineral sulfonate 505

Oil, mineral, with glyceryl monooleate or glyceryl naphthenate 506, 507

Oil, mineral, with glycerylmonooleate and mahogany soap 508

Oil, mineral, with gum acacia and tragacanth or agar 509

Oil, mineral, with heptadecylamine hydrochloride and trisodium phosphate, sodium sulfate, palmitic acid, sodium tannate or 3N hydrochloric acid 510

Oil, mineral, with lignosulfonic acid 511

Oil, mineral, with lime water (calcium hydroxide sol'n.) 512

Oil, mineral, with (skim) milk or diglycol laurate 513

Oil, mineral, with monostearin and sodium stearate 514

Oil, mineral, with montan wax 515

Oil, mineral, with naphthalene sulfonic acid 516

Oil, mineral with naphtylamine or quinol 517, 518

Oil, mineral, with nonaethylene glycol dioleate 519

Oil, mineral, with oxidized naphthenic hydrocarbons 520

Oil, mineral, with petroleum sulfonates (water soluble)<sup>521</sup>

Oil, mineral, with phosphorized and sulfurized lard oil. Twitchell reagent and diethylene glycol 522

Oil, mineral, with polyhydric alcohols partially esterified with higher fatty acids 523

Oil, mineral, with polymerized oil ("Voltol"), wool fat or wool fat alcohols 524

Oil, mineral, with saponified sulfonated oil soap 525

Oil, mineral, with saponified sulfonated sperm oil 525

Oil, mineral, with sodium cetyl sulfate, cholesterol, oleyl alcohol, elaidyl alcohol, cetyl alcohol, sodium stearate, sodium elaidate, sodium oleate, cetyl sulfate, cetyl sulfonate, sodium palmitate, cetyltrimethyl ammonium bromide, sodium taurocholate, sodium glycocholate or sodium desoxycholate 526

Oil, mineral, with sodium oleate, arabic acid, sodium, magnesium or iron arabate, sodium or ammonium valerate, sodium galactonate, sodium dihydroxy stearate, sodium salicylate or sodium gallate 527

Oil, mineral, with sodium oleate or haemoglobin 528

Oil, mineral, with sodium oleate or Turkey red oil 529

Oil, mineral, with sodium petroleum sulfonate and sodium rosinate  $^{530}$ 

Oil, mineral, with sodium salt of sulphated fish oil 531

Oil, mineral, with sodium, and triethanolamine naphthenates 532

Oil, mineral, with soya bean albumin and soda ash 533

Oil, mineral, with starch, dextrin, and flour or gum senegal, or gum arabic with gum tragacanth or karaya 584

Oil, mineral, with sulfonated castor oil or caustic alkali and olein, rosin or rosin oil, ammonia and alcohol 535

Oil, mineral, with sulfonated castor oil, neutralized (Turkey red oil) 536

Oil, mineral, with sulfonated fatty alcohol salt 537

Oil, mineral, with triethanolamine sulfonate 588

Oil, mineral and acetic acid (glacial), with "Emulgor A" and oleic acid 589

Oil, mineral and aluminum chloride, with "Emulgor A" and oleic acid 540

Oils, mineral and animal, with sulfurized unsaturated oil 541

Oil, mineral, animal or vegetable, with lead salt of sulfated higher fatty alcohol  $^{542}$ 

Oil, mineral, benzene or olive oil, with sodium oleate, saponin or triethanolamine oleate 543

Oil, mineral, butanol, with naphthenic soap 544

Oil, mineral, and but anol, with sulfonated oleic acid sodium salt  $^{545}\,$ 

Oil, mineral, castor or olive, with saponin or sodium oleate, or gum tragacanth or sodium glycocholate or gum acacia 546

Oil, mineral and citric acid, with "Emulgor A" and oleic acid 547

Oil, mineral, creosote, with oil soluble mineral oil sulfonates 548

Oil, mineral, and cresol, with fish oil soap 549

Oil, mineral, and cresol, with sodium naphthenate 550

Oil, mineral, decalin, olein cyclohexanol and turpentine, with soap 551

Oil, mineral, and dinitro-o-cyclo-hexylphenol, with blood albumin  $^{552}$ 

Oil, mineral, fatty acid, carbon disulfide, turpentine or chlorinated hydrocarbons, with olein, soap and cyclohexanol <sup>558</sup>

Oil, mineral, fatty acids (vegetable or animal) and gas oil, with castor oil soap  $^{554}$ 

Oils, mineral and fish, with ammonium soap 555

Oil, mineral and formic acid, with "Emulgor A", oleic acid and glaurin 556

Oil, mineral, and hexalin, with mahogany sulfonates and gelatin  $^{557}$ 

Oil, mineral, and hydrocarbon gas, with bentonite 558

Oil, mineral and hydrochloric acid, with "Emulgor A" and glaurin 559

Oil, mustard or lemon, with gum acacia 560

Oil, mineral or mustard, with pectin 561

Oil, mineral, and naphthenic acid, with sulfonated naphthenic acid  $^{562}$ 

Oil, mineral, nicotine petroleum sulfonate, with soap 568

Oil, mineral, and nicotine sulfate, with bentonite or diglycol oleate 564

Oil, mineral, and oleic acid, with lime and soda ash 565

Oil, mineral, and oleic acid, with sodium salts of petroleum sulfonates,  $^{566}$ 

Oil, mineral, and oleic acid or naphthenic acid or alcohol rosin solution, with sodium petroleum sulfonates 567

Oils, mineral and olive, with soap and sulfonated castor oil <sup>568</sup> Oil, mineral, olive or castor, with sulfonated higher fatty alcohol <sup>569</sup>

Oil, mineral or olive, or oleine, with "Nekal A.E.M." 570

Oil, mineral, or other hydrocarbons and alcohol, with nascent hydrogen <sup>571</sup>

Oil, mineral, and oxidized mineral oil, with potash soap 572

Oil, mineral and oxyquinoline sulfate, with "Emulgor A" and oleic acid 578

Oil, mineral, and phenol, with sodium resinate 574

Oil, mineral and phosphoric acid, with "Emulgor A" oleic acid and glaurin 575

Oils, mineral and pine, and turpentine, with calcium soap 576

Oil, mineral, and rosin, with catechu or other tannin 577

Oil, mineral and sodium chloride, with "Emulgor A" 578

Oil, mineral and sulfuric acid (conc.), with "Emulgor A", oleic acid and glaurin 579

Oils, mineral and tall, with alkali 580

Oil, mineral or tar, with colloidal graphite, magnesium, oxide or carbonate 581

Oil, mineral or tar, with glue and urea 582

Oil, mineral or tar, with sulfite lye and caustic potash 583

Oils, mineral and tar-acid, and isopropanol, with oil soluble petroleum sulfonate 584

Oil, mineral and tartaric acid, with "Emulgor A" and oleic acid 355

Oil, mineral and terpinyl acetate, with soap 586

Oil, mineral, and turpentine, with antimony chloride 587

Oils, mineral and vegetable, with soap and sulfonated oil 588

Oil, neatsfoot, with aluminum hydroxide 589

Oil, neatsfoot, with diethyloctylamine hydrochloride 590

Oil, neatsfoot, with sulfonated cod oil and soap 591

Oil, neatsfoot, with sulfonated neatsfoot oil and egg yolk or egg white 592

Oils, neatsfoot and mineral, with sulfonated neatsfoot oil  $^{598}$  Oil, neatsfoot, train, bone, or rapeseed, with methyl cellulose  $^{594}$ 

()il, olive, with ammonium oleate 595

Oil, olive, with casein 596

Oil, olive, with (alkaline) casein 597

Oil, olive, with castor oil soap 598

Oil, olive, with (skim) milk 599

Oil, olive, with milk and egg yolk or starch 600

Oil, olive, with oleyldiethylethylene diamine hydrochloride 801

Oil, olive, with sodium oleate 602

Oil. olive, and beef suet, with Irish moss 603

Oil, olive or castor, with colloidal clay and alkali 604

Oil, olive, castor, cedar or cod liver, with medicinal plant infusions, e.g., absinth or mint 605

Oil, olive, castor, linseed or mineral, oleic acid, paraffin wax, higher alcohols, ketones or esters, with soap and glue, gelatin or gum arabic, in place of the soap, saponin or concentrated sulfite liquor can be used 606

Oil, olive or cod liver, with sodium silicate 607

Oil, olive, and paraffin wax, with glue 608

Oil, olive, peanut, or cottonseed, with alkaline casein 609

Oil, olive, peanut, cottonseed, or soya bean, lard, oleo or hy-

drogenated oils (castor, cod liver, or mineral oil), with raw egg, egg-white, egg yolk or dried egg 610, 611

Oil, olive, peanut or other oils, with sulfonated oils, fats or fatty acids 612

Oil, olive, sperm, castor, poppy or cod liver, with casein 613

Oil, olive or sweet almond, with gum tragacanth or gum arabic 614

Oil, olive, and tallow, with potassium carbonate 615

Oil, oxidized drying, with sodium hydroxide or ammonia 616

Oil, palm, with Duponol ME, cetyl alcohol and borax 617

Oil, palm, with triethanolamine and tall oil 618

Oils, palm and mineral, with soap 619

Oil, palm and tallow or rapeseed oil, with sodium carbonate 620

Oil, paraffin, with gelatin or Sapamine MS 621

Oils, paraffin and fish, with soap and ammonia 622

Oil, paraffin, benzene, nonyl alcohol or undecyl alcohol, with sodium oleate 628

Oil, paraffin or cod liver, with "Tylose" (methyl cellulose) 624 Oils, paraffin and linseed, turpentine and ether, with ammonia 625

Oil, peanut, with ammonia 626

Oil, peanut, with egg white and milk 627

Oil, (hardened) peanut, with milk and egg yolk proteins<sup>628</sup>

Oil, peanut, with oleyl or cetyl adipate 629

Oil, pine, with alkali and amine soap 630

Oil, pine, with alkali soap and amine soap 630

Oil, pine, with soya bean flour and soap 681

Oil, pine and hydrochloric acid, with "Emulgor A" and oleic acid  $^{632}$ 

Oil, pine and sodium chloride, with "Emulgor A" 682

Oil, pupa, with sodium oleate, gum arabic or dextrin 633

Oil, rapeseed, with milk curds 634

Oil, rapeseed, turpentine, oil of amber and camphor with soft soap and ammonia 635

Oil or resin with glue or egg yolk 636

Oil, sardine, with sulfonated naphthenic acid salt 637

Oil, sesame, with polyglyceryl oleate 638

Oil, shale, with lime 639

Oil, shale distillate, with sulfite liquor, soap, glue, calcium cascinate or bentonite  $^{640}$ 

Oil, soya bean, with caustic and alum 641

Oil, sperm, with glyceryl monostearate and sodium oleyl sulfonate  $^{642}$ 

Oil, sulfurized, with gelatin, casein, soap or saponin 643

Oil, sulfurized colza, with saponin or soap 644

Oil, tall, with soap and aniline 845

Oil, tar, asphalt or rubber, with aromatic or hydroaromatic sulfonic acids or their alkali, calcium or magnesium salts 646

Oil, tar, asphalt or rubber, with sodium petroleum sulfonates 647

Oil, ti-tree, with cetyl alcohol 648

Oil, train, neatsfoot, or mineral, or wool fat with methyl cellulose 649

 $\mathrm{Oil},$  turpentine, with gelatin, water, glycerin, sucrose and tartaric acid  $^{650}$ 

Oil, vegetable, with ammonia and insecticide 651

Oil, vegetable, with casein and alkali or amine 652

Oil, vegetable, with egg yolk 653

Oil, vegetable, with glycerylmono and distearates 654

Oil, vegetable, with lecithin and alkali peroxide 455

Oil, vegetable, with (skim) milk 656

Oil, vegetable, with milk and partially oxidized soya bean oil  $^{657}$ 

Oil, vegetable, with polyhidric alcohol partially esterified with higher fatty acid  $^{658}$ 

Oil, vegetable, with sodium potassium or ammonium soaps 659

Oil, vegetable, with starch and gum arabic or tragacanth 660

Oil, vegetable, or fat, with pectin 661

Oil, vegetable, or fatty acid, with vegetable phosphatides 862

Oil, vegetable, fish or mineral, and boiled linseed oil, with maltose 603

Oil, vitamin, concentrate, with gum arabic, pectin or agar 664

Oil, volatile, creosote or thymol, with rosin soap 665

Oil, waxes or resins, with ammonium linoleate 666

Oleic acid, with ammonium oleate 667

Oleic acid, with sodium oleate 668

Oleic acid, with triethanolamine salt of oleyl sulfate 669

Oleic acid, cyclohexanol, aniline or benzol, with ditolyl sodium phosphate 670

Olein, with cetyl glucoside and Turkey-red oil 671

Olein, with methyl cellulose and sulfated lauryl alcohol 672 Paraffin wax, with calcium oleate 673

Paraffin wax, with diethylamine oleate 674

Paraffin wax, with (higher) fatty acid amide 675

Paraffin wax, with gelatin 676

Paraffin wax, with glue, alumina-gel, gelatin, soap 677, 678, 679, 680

Paraffin wax, with gum ghatti 681

Paraffin wax, with gum tragacanth 682

Paraffin wax, with rubber latex 683

Paraffin wax, with saponified montan wax 684

Paraffin wax, with soap 685

Paraffin wax, with sodium salt of petroleum sulfonic acid 686

Paraffin wax, with sodium soaps of stearic, palmitic, ricinoleic or cerotic acid 687

Paraffin wax, with soybean protein 688

Paraffin wax, with sulfite cellulose extract and clay 689

Paraffin wax, with trihydroxyethylamine stearate 690

Paraffin wax and aluminum acetate, with "Emulgor A", oleic acid and glaurin 691

Paraffin and beeswax and gum elemi, with sodium hydroxide 692 Paraffin wax, beeswax, tallow, olive-oil or oleic acid with Lamepone A 693

Paraffin wax and benzene, with soap 694

Paraffin wax and benzol with alkali casein and rosin 695

Paraffin wax, bitumen, oil or fat, with degraded starch 696

Paraffin wax and carbon tetrachloride, with gelatin, glue or gum tragacanth  $^{697}$ 

Paraffin or ceresin wax, with soap and glue, albumin, casein, China clay or amines 698

Paraffin, esparto, candelilla, wool, or montan wax and rosin with caustic soda 699

Paraffin wax and esparto, wool or montan wax, with rosin soap  $^{700}$ 

Paraffin wax and linseed oil, with albumin 701

Paraffin or montan wax, with corn oil soap and starch <sup>702</sup> Paraffin wax and montan wax, with rosin soap and alkali <sup>703</sup>

Paraffin, montan, ceresin, spermaceti, or ozokerite wax, with

cetyl alcohol, wool fat alcohol, mono- or di-glycerides with or without soap  $^{704}$ 

Paraffin wax and oil, with alkali fatty acid soap and glue 705 Paraffin wax or oil, with sodium dodecyl sulfate and lauryl pyridinium bisulfate 706

Paraffin wax and phenol-formaldehyde resin, with sulfonated sperm oil and soap 707

Paraffin wax or resins, with glue or starch 708

Paraffin wax and rosin, with rosin soap 709

Paraffin wax and shellac, with soap 710

Paraffin wax and wool grease, with blood albumin 711

Peat, with calcium oxide 712

Petrolatum, with agar 713

Petrolatum, with Turkey red oil 714

Petrolatum, with wool wax 715

Petrolatum and beeswax or stearic acid, with sodium salt of sulfonated hydrogenated castor oil or sulfonated castor oil 716

Petrolatum and ceresin, with slaked lime 717

Petrolatum or fat with phytosterols 718

Petrolatum and tallow, with Turkey red oil soap 719

Petroleum (cylinder), with calcium oxide 720

Petroleum, with casein 721

Petroleum, with ethanolamine dihydroxystearic acid 722

Petroleum, with oil soluble sulphonated naphthenic acid 723

Petroleum, with sodium petroleum sulfonate 724

Petroleum, with sulfonated peanut oil and copper soap <sup>725</sup> Petroleum, with sulfonated sodium salt <sup>726</sup>

Petroleum or coal tar pitch, with ammonium alginate 727

Petroleum distillate, with condensation product of sulfonated oil and glycol or glycerol 728

Petroleum, and moellon, with wool grease and soap 729
Petroleum, pitch or bitumen, with sodium chloride or glycerol 730

Petroleum residue, with starch <sup>731</sup>

Petroleum residue or gasoline, with sea water 732

Petroleum, turpentine, chloroform, ethyl chloride ethyl bromide, ionone, terpineol, nitrobenzene aldehydes, pyridic or quinolic bases, tar, essential oils, bone oil, phenols, or naphthols, with sodium soap of castor oil 733

Phenanthrene, retene, picene or chrysene, halogenated, with

soap, amine soap, gum arabic, starch, bentonite saponified montan wax or casein 784

Phenol or cresol, with sodium sulfonates of shale tar 785

Phenol and toluol, with sodium oleate 736

Pitch, with bentonite 737

Pitch, with chrome green 738

Pitch, with soap and alkaline casein 739

Pitch, with starch and tannic acid or quebracho 740

Pitch, asphalt, resin or rubber, with bentonite 741

Pitch, coal-tar, with ammonia 742

Pitch, coal-tar, or asphalt, with clay 748

Pitch, stearin and bitumen, with caustic soda or potash 744

Pitch or tar, with sodium metasilicate 745

Polyisobutylene and carbon tetrachloride, with sodium oleate and casein 746

Polyvinyl compounds, with rosin soap 747

Polyvinylisobutyl ether or polyvinylethylether, with ammonia and casein 748

Pyrethrum extract, with sulfonated aromatic compounds <sup>749</sup> Pyrethrum solution, with triethanolamine abietate <sup>750</sup>

Resin, with alkaline casein 751

Resin, alkyd, with ammonia and casein 752

Resin, alkyd, with ammonium diisopropyl naphthalene sulfonic acid  $^{753}$ 

Resin, alkyd, with casein and alkalies 754

Resin, alkyd, with inorganic alkalies or organic bases 755

Resins, alkyd and maleic with casein, ammonia and montan wax <sup>756</sup>

Resin, cellulose ester, with gum ghatti and sodium isopropylnaphthalene sulfonate  $^{757}$ 

Resin, cumarone, with sodium silicate 758

Resin, cumarone, with triethanolamine and oleic acid 759

Resin, cumarone-indene, with sodium silicate 760

Resin, cumarone, and solvent, with Turkey red oil 761

Resin, ethyl acrylate or styrene, with sodium isopropyl naphthalene sulfonate  $^{762}$ 

Resin, ethyl or benzyl cellulose, and solvent, with alkaline casein and sulfonated vegetable oil 763

Resin "Glyptal" solvent and rubber, with bentonite and soap 764

Resin, ethylcellulose, butyl acetate octyl acetate and toluol, with Deceresol O.T. (10%)<sup>765</sup>

Resin, methyl acrylate, with sodium isopropyl-naphthalene sulfonate or sulfonated castor oil  $^{766, 767}$ 

Resins, methyl-methacrylate and allyl-methacrylate, with coconut oil soap  $^{768}$ 

Resins, methyl-methacrylate, butyl-methacrylate, dammar and dibutyl-phthalate, with sodium decanolsulfate 769

Resin, Neville, with triethanolamine oleate and trisodium phosphate 770

Resin or oil with dry precipitated silicic acid 771

Resin, paracumarone, with sodium silicate 772, 773

Resin, phenol-formaldehyde, with ammonia casein and methylcyclohexanol stearate 77\*

Resin, phenol-formaldehyde (partially reacted), with casein and alkali <sup>775</sup>

Resin, phenol-formaldehyde, with formaldehyde-phenol sulfonic acid 776

Resin, phenol-formaldehyde, with gum ghatti or arabic 777

Resin, phenol-formal dehyde, with sodium hydroxide and gum arabic  $^{778}$ 

Resin, phenol-formal dehyde, and benzene, with bentonite and sodium oleate  $^{779}\,$ 

Resin, polyvinyl acetate, with polyglycol laurate 780

Resin, synthetic, e.g., tung oil and maleic anhydride resin, with ammonia, sulfonated oils, soaps or wool-fat acids and gum tragacanth or colloidal clay 781

Resins or terpenes, with ammonium sulforicinoleate 782, 783 Resin, thermoplastic, with soap 784

Resin, urea-formaldehyde, with sulfated ricinoleic acid and naphthalene salts 785

Resin, vinyl, with glue 788

Resin, vinyl copolymer, and benzol, with Alkanol B 787

Resin, vinyl, and toluene, with glue 788

Rosin, with mono-, di-, or tri-hydroxyethylamine or hydroxyethyl alkylamines 789

Rosin, with isobutylnaphthalenesulfonic acid 790

Rosin, with rosin soap 791, 792

Rosin, with soda ash 798

Rosin, with soda ash, sodium bicarbonate and ammonia 794

Rosin, with sodium caseinate 795

Rosin, with sodium hydroxide 796

Rosin, with sodium-, potassium-, or ammonium-, lauryl, cetyl, or octadecyl sulfate  $^{797}$ 

Rosin acids, with glue or gum arabic 798 Rosin and benzine, with rosin soap 799

Rosin, carbon tetrachloride, mineral oil, cresol, with rosin and caustic soda 800

Rosin and essential oils, with rosin soap 801

Rosin and lubricating oil, with alkaline casein 802

Rosin and paraffin wax, with casein and starch 803

Rosin and paraffin wax, mineral oil, tar oil, fatty oil, montan wax, turpentine, pine oil, butyl oleate or ethyl stearate with soap or alkaline casein 804

Rosin and paraffin wax or turpentine, with casein and soap <sup>805</sup> Rosin, rubber and cymene, with caustic soda <sup>806</sup>

Rosin and shellac, with caustic soda and soap 807

Rosin, turpentine, and Japan wax, with borax and soap 808 Rubber, with bentonite 809, 810

Rubber, with calcium oleate 811

Rubber, with casein 812

Rubber, with colloidal clay 813

Rubber, with glue 814

Rubber, with glue, albumin, casein, gum arabic, sugars, gluten, starch, dextrin or colloidal clay and soap or saponin 815

Rubber, with glue, casein or gluten 816

Rubber, with glue, soap and alkali casein 817

Rubber (vulcanized), with glue, soap or casein 818

Rubber, with morpholine soap 819

Rubber, with oilseed cake and hulls and alkali 820

Rubber, with soap 821

Rubber, with soap, colloidal clay, sea moss or albuminate or gelatinate 822

Rubber, with soap, proteins, etc.828

Rubber, with sodium abietate, rosinate, oleate, palmitate or stearate  $^{824}$ 

Rubber, with sodium cetyl sulfate 825

Rubber (plasticized), with sodium silicate 826

Rubber, with sodium toluene sulfonate, potassium hydroxy-

naphthoate, potassium sulforicinoleate, potassium sulfanilate or sodium cholate  $^{827}$ 

Rubber, with sulfonated mineral oil 828

Rubber, balata or gutta-percha, benzene and sulfur, with ammonium oleate 829

Rubber and benzene, with soap 830

Rubber and benzene, with soap and ammonium carbonate <sup>831</sup> Rubber, benzene and kerosene, with sulfonated castor oil soap <sup>832</sup>

Rubber chloride solution, with dextrin, casein, starch or gum arabic  $^{833}$ 

Rubber, chlorinated, and carbon tetrachloride, with an alkali s34 Rubber, chlorinated, cellulose esters or cellulose ethers and a solvent, with ammonium or potassium oleate s35

Rubber, chlorinated, and trichlorethylene, with alkaline casein and Turkey-red oil 886

Rubber and cumar or rosin, with whiting and rosin soap, locust-bean gum, starch or dextrin 887

Rubber and cymene, with rosin soap 888

Rubber, fluorinated, and solvent, with sodium stearate 839

Rubber and gasoline, with ammonium sulforicinoleate 840

Rubber (raw or vulcanized), gutta percha, balata or resins, with ammonium oleate 841

Rubber and naphtha or benzol, with ammonium oleate 842

Rubber and mineral oil, with casein and ammonia soap 843 Rubber and oil, with metallic soap 844

Rubber and oil, with sodium alkylnaphthalene sulfonate 845

Rubber, reclaimed, with glue, soap, casein, gelatin or saponin 846

Rubber, reclaimed, cumar and rosin oil, with sodium hydroxide 847

Rubber, reclaimed, and spindle oil, with rosin soap or sulfonated castor oil 848

Rubber and rosin, with alkali 849

Rubber and solvent, with ammonia soap 850

Rubber and solvent, with saponin 851

Rubber, tar. rosin and solvent with alum and lime 852

Rubber and turpentine, with glue and sodium carbonate 853

Rubber and varnish with soap 854

Sodium butadiene rubber with ammonia soap and casein 855

Solvent (naphtha) with benzyl alcohol and butylnaphthalene sulfonic acid  $^{856}$ 

Solvents with carragheen kelp extract and oxalic acid 857

Solvent, high boiling with bentonite and soap 858. 859. 860

Solvent and phenol with tall oil and alkali 861

Shale asphalt and montan wax with soap 862

Shellac with protective colloids 863

Soya protein and paraffin wax, with ammonium oleate 864

Spermaceti, with borax and gum arabic 865

Stearic acid, with ammonia, caustic potash, borax, triethanolamine, sulfonated oils, vegetable gums or glycol- or glyceryl stearates see

Stearic acid, with glue and sulfonated castor oil 867

Stearic acid with sodium salt of naphthalene sulfonate 868

Stearic acid, hydrogenated oils, waxes, mineral or fatty oils, with sulfonated higher fatty alcohols or hydrocarbons, e.g., lauryl alcohol sulfate 869

Stearic acid or other higher fatty acid and paraffin oil or wax, with albumin or globulin 870

Stearic acid, spermaceti and cetyl alcohol, with caustic soap  $^{\rm 871}$ 

Stearic anhydride, with deacetylated chitin 872

Stearic anhydride, with soap 873

Styrene, with ammonia soaps 874

Styrene, with ammonium oleate or saponin, sulfonates or albuminoids 875, 876

Styrene with soap or cetylpyridinium bromide 877

Sulfur, with agar or carragheen 878

Sulfur and asphalt, with sodium oleyl sulfate 879

Sulfurized fatty oils, with tannic orgallic acid or tannins 850 Sulfurized linseed oil, with tannin, gallic or tannic acid 880

Tallow or beef fat and butter or vegetable oil, with skim milk 881

Tallow and beeswax, with caustic potash 882

Tallow and cresol, with soap 883

Tar, with cumarone resin or tosin with soap ss4

Tar, with kaolin and borates, fluorides and chlorides 885

Tar, with lime and aluminum salts 886

Tar, with rosin soap and flour 887

Tar, with saccharates of sodium, potassium, calcium, strontium or barium \*\*\*ss\*\*

Tar, with soap and protein 889

Tar, with sodium hydroxide 890

Tar or asphalt, with soap and higher monohydric, aliphatic alcohol 891

Tar or asphalt, with sodium petroleum (green) sulfonates 892 Tar, asphalt or pitch, with metallic soap 893

Tar or balsams, with egg yolk 894

Tar or bitumen, with starch and soap 895

Tar, coal, with polyvinyl chloride, soap, alkali protein or rosin soaps 896

Tar, coal, with soap and tar 897

Tar, coal, with soda ash and casein 898

Tar, coal, with sodium oleate 899

Tar, coal, with tincture of quillaja 900

Tar, coal, and asphalt flux oil, with ammonium oleate 901

Tar, coal or bitumen, with colloidal clay, sea-weed extract, soap, casein, starch, glue or gums 902

Tar, coal, and mineral oils, mineral wax, naphthalene, or pitch, with alkali alginate <sup>908</sup>

Tar, coal, mineral, vegetable or animal oil with glue and rosin soap 904

Tar, coal, and stearin pitch with caustic potash and casein 905 Tar, coal or wood, with sulfonated lignin sodium salt 906

Tar, creosote, bitumen, or petroleum with rosin soap and protein  $^{907}$ 

Tar and pepper oil with soap 908

Tar or petroleum pitch with clay 909

Tar, pitch or bitumen with rosin soap 910

Tar, tar oil, petroleum or asphalt with brown coal 911

Tar, wood, with algin 912

Tar, wood, with soap and Irish moss 913

Tar, wood or coal, or mineral oil residues with concentrated spruce sulfite liquor 914

Terpene, maleic anhydride, polyhydric alcohol resin, with sodium hydroxide 915

Terpineol, with sodium abeitene-sulfonate 916

Tetrachlorethane, with laurylamine hydrochloride 917

Tetrachlorethylene, with rosin soap and alkali casein 918

Tetralin, with sodium oleate 919

Thiuram and coconut oil, with casein 920

Toluol, with sulfonated castor oil or sodium oleate streight 921 Toluol and hydrochloric acid, with "Emulgor A" and oleic acid 922

Toluol and sodium chloride, with "Emulgor A" and oleic acid 922

Triazine (melamine) resins, with soap, or sulfated fatty alcohol 923

Trichlorethylene with alcoholic sodium oleate 924

Trichlorethylene with bentonite and Turkey red oil 925

Triglycerides, with lecithin or polyhydroxy alcohols partially esterified with higher fatty acid 926

Turpentine, with gelatin or egg albumin 927

Turpentine, with pitch rosin soap 928

Turpentine, with soap 929

Turpentine, oil of thyme, oil of amber and petroleum, with soap and caustic potash 980

Turpentine and pine oil, with oxgall 931

Turpentine and suet, with soap 932

Varnish, with ammonium soaps 933

Varnish, with gum tragacanth 934

Varnish, with mahogany petroleum sulfonates 935

Varnish, with soap and glue 986

Varnish, with soap or sulfonated oils 987

Varnish (Japan), with sodium hydroxide 938

Varnish, with soybean protein and ammonia 989

Varnish, with saponified montan wax 940

Varnish and solvents, with sodium stearate 941

Varnish (Japan), or asphalt with soap or alkali casein 942

Vinyl acetate, with methyl cellulose or ethers or acetals of polyvinyl alcohol 948

Vinyl acetate, with sulfonated castor oil 944

Vistanex polybutene and solvents, with soap, casein or gums <sup>945</sup> Vitamins (oil soluble), with fatty acid ethers or esters of polyhydroxy alcohols <sup>946</sup>

Vitamin A and D concentrates, with morpholine oleate, ricinoleate or linoleate 947

Vitamin D concentrate, with polyglyceryl partial fatty acid ester, lecithin or glyceryl monolaurate 948

Vitamin containing vegetable oils, with agar or Irish moss or egg yolk 949

Vitamins, or irradiated ergosterol and vegetable oil, with gelatin, agar, carragheen, egg yolk, casein, gum arabic or tragacanth 950

Wax, with cetylmethylpiperidinium methylsulfate 951

Wax, with soap 952

Wax, hydrocarbon, with oil soluble petroleum sulfonic acid soap 953

Wax, hydrocarbon, or oils, with alkylated naphthalene sulfonic acid or salt 954

Wax or rosin and coal oil, with bentonite 955

Wax, vegetable, with caustic potash 956

Wool fat, with methyl cellulose 957

Wool fat and varnish resin, with ammonia soap 958

Wool grease, with blood albumin 959

Wool grease, with cyclohexanol 960

Xylene, with sodium oleate and phenol 961

Xylene and phenol, with sodium oleate 962

Xylene or sulfonated xylene, with Turkey red oil or soap or sulfated higher alcohol salts  $^{963}$ 

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#### CHAPTER IX

# LIST OF DEMULSIFYING AND DEFOAMING AGENTS

Acetylated hydroxy higher fatty acid is used for demulsifying petroleum.<sup>1</sup>

Acetylsulforicinoleic acid is used as a demulsifier.2

Acylated amino ether is used as a demulsifier.3

Ammonium sulfide is a demulsifier.4

Amylated aromatics are used as demulsifiers.5

Break the petroleum emulsion, by passing under pressure through cloth or filter earth.6

Calcium acetate with monohydric or dihydric alcohol is used as a defoamer.<sup>7</sup>

Combined magnetic and electric fields are used for demulsifying.8

Condensation product of sulfonated aniline and aliphatic alcohol or aldehyde acts as demulsifier.<sup>9</sup>

Degras is dispersed under pressure into fermentation tank to break Brewer's foam.<sup>10</sup>

Electrical breaking of emulsion is accomplished by passing between high tension electrodes. 11, 12, 13, 14, 15, 16, 17, 18, 19, 20

Electrical and mechanical baffle effects are used for demulsifying petroleum.<sup>21</sup>

Esters of blown fatty oils are used for breaking petroleum emulsions.<sup>22</sup>

Ester of polybasic carboxylic acid and an acylated amino ether are used as demulsifiers.<sup>23</sup>

Glyceryl monoricinoleate is used for demulsifying petroleum.<sup>24</sup> Gravity flow is used to break crude oil emulsions.<sup>25</sup>

Halogenated sulfonated fatty acid or ester is used for demulsifying petroleum.<sup>26</sup>

Heating with SO<sub>2</sub> and H<sub>2</sub>S breaks emulsions.<sup>27</sup>

Heat petroleum emulsions and pass them under pressure through infusorial earth, filter cloth and fine metal screen.<sup>28</sup>

Heat and vacuum are used to break foam. 29

Intermittent direct current is superimposed on the applied alternating current at intervals of several seconds for breaking petroleum emulsions.<sup>30</sup>

Inversion of W-O emulsions, by reagents, e.g., by oxidized hydrocarbons, often breaks emulsions.<sup>31</sup>

Isoamyl valerate is a defoamer.82

Isopropanal and tributyl phosphate are used as a defoamer.<sup>33</sup> Kerosene emulsions are broken by carbon black.<sup>34</sup>

Laurylamine hydrochloride is a defoamer.85

Lignin sulfonate and propylnaphthalene sulfonic acid are demulsifiers.<sup>36</sup>

Lower fatty acid esters of hexyl, heptyl or octyl alcohols are foam inhibitors.<sup>87</sup>

Microbes and sustaining medium break emulsions.38

Passing between moving gears of different wettabilities is used for breaking emulsions.<sup>39</sup>

Passing through capillary media, e.g., cloth is used for demulsifying.<sup>39a</sup>

Passing through zeolites and iron sulfide and quartz breaks emulsions.<sup>40, 40a</sup>

Phosphatide (lecithin) is a foam reducer.41

Reaction product of abietine, aldehyde and sulfonating agent are used for breaking emulsions.<sup>42</sup>

Reaction product of blown fatty oil with aldehyde reactive agent, used for demulsifying petroleum.43

Reaction products of sulfonated cottonseed oil with chlorinated cyclopentane are used to break mineral oil emulsions.<sup>44</sup>

Sodium chloride and magnesium oxide or "Tret-o-Lite" (patented mixture of soap, sodium silicate, phenol, paraffin and water.); ether and carbon bisulfide are used for breaking emulsions. 45, 46

Sodium sulfobenzyl phthaloricinoleate is a demulsifier.47

Solid aliphatic alcohol and liquid aliphatic alcohol of at least 8 carbon atoms, used as a defoamer. 48

Sugars break certain oil emulsions by dehydration at the interface.49

Sulfated cetyl alcohol is a demulsifier.50

Sulfonated fatty glyceride and calcium chloride are used in breaking emulsions.<sup>51</sup>

Sulfated lauryl alcohol is used for breaking emulsions.<sup>52</sup>

Sulfonated mixture of rosin oil and castor oil, used for demulsifying petroleum.<sup>58</sup>

Tar oil emulsions containing sodium phenolate are resolved by allowing to stand.<sup>54</sup>

Violet, ultra-violet light, X-ray etc. are used for breaking emulsions.<sup>55, 56, 56a</sup>

Water insoluble basic alkyl salt of petroleum sulfonic acid is used for breaking petroleum emulsions.<sup>57, 58</sup>

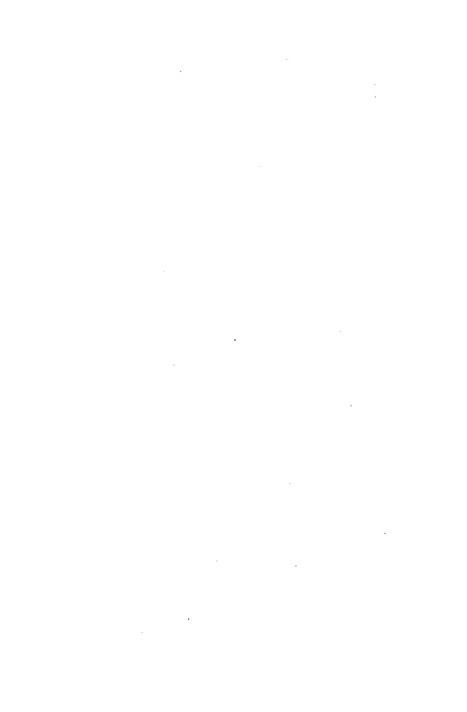
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# PART II FORMULAS



#### CHAPTER I

# AGRICULTURAL SPRAY EMULSIONS

# Formula No. 1.

Mineral Oil 2-2 $\frac{1}{2}$  gal. Trihydroxyethylamine Diglycol Oleate 2-3 oz. Linoleate  $\frac{1}{2}$ -1 lb. Water 100 gal.

Add in the above order and beat vigorously. The above emulsion is quick breaking and spreads easily.<sup>1</sup>

#### FORMULA No. 2.

A petroleum fraction having a sulphonation value above 50, 1.2–0.3 per cent, trichlorobenzene 0.4–0.1 per cent, an emulsifying agent 0.04–0.01 per cent, water 98.36–99.59 per cent.<sup>2</sup>

#### FORMULA No. 3.

Mineral Oil (60-80 sec.)	95.95	Beta Naphthol	0.05
Aluminum Oleate	2.50	Water 3	To suit
Glyceryl Monooleate	1.50		

#### FORMULA No. 4.

Anthracene Oil Fish Oil Soap	75 <b>3</b>	Water 4	22
rish Oil Soap	J		

<sup>&</sup>lt;sup>1</sup> Bennett, Chem. Formulary, Vol. 1, p. 215 (1933).

<sup>&</sup>lt;sup>2</sup> U. S. 2,046,901.

<sup>&</sup>lt;sup>3</sup> Sharp, U. S. 2,240,806 (1941).

<sup>4</sup> Bennett, Chem. Formulary, Vol. 1, p. 215 (1933).

#### FORMULA No. 5.

Diglycol Laurate Pyrethrum Extract	5 qt. (20 Fold Water 5	1) 3/4 p 100 gal.
	FORMULA No. 6.	
Derris Extract (5%) Skim Milk Powder	1 qt. Water 6	100 gal.

#### FORMULA No. 7.

Mineral Oil 93 Nicotine Oleate Glyceryl Oleate 2

0.84 pounds of casein is slowly poured into about 2 gallons of cold water, and stirred until thoroughly wet and soaked, then 0.63 pounds of dehydrated sodium carbonate is added, stirring until all the casein is well in solution. Six gallons of denatured alcohol is then added, and 1.67 pounds of powdered gamboge. The gamboge is added slowly with constant stirring. Enough water is then added to make up a total of 20 gallons. With this composition, the oil to be emulsified is incorporated, preferably by slow additions, with agitation. Most oils emulsify therein readily. Heat may be applied if quicker emulsification is desired. For petroleum oil for example, with a specific gravity of 0.891 a proportion of 1 part by volume of the foregoing composition to 5 parts of the oil affords a satisfactory product. Such emulsion will contain about 83.3% of oil, making up to a consistency about that of lard at the same temperature. Such a product even after standing in a warm place for months is free from separation.7

#### FORMULA No. 8.

Mineral Oil	91.4	Nicotine	2.0
Glyceryl Oleate	1.5	Napthenic Acid	3.1
Aluminum Oleate	2.0	Add water before use.	

<sup>&</sup>lt;sup>5</sup> Ihid., Vol. 3, p. 119 (1936).

<sup>6</sup> Ibid.

<sup>&</sup>lt;sup>7</sup> Bennett, Chem, Formulary, Vol. 1, p. 216 (1933).

Nicotine, commercial (95%), 5 fl. oz.; soft soap, commercial, 2½ lb.; alcohol (95%), 80 fl. oz. For use, dilute with 160 volumes of water. A mildew spray is: Linseed oil, 12 fl. oz.; liver of sulphur, 2 lb.; cresol, 3 lb.; caustic potash, commercial, 1 lb.; water to make 1 gallon.8

	FORMULA	No. 9.	
Olein Coal Tar Oil	20 kg. 70 kg.	Caustic Potash (24° Bé.)9	19 kg.
	FORMULA	No. 10.	
Talloil, Crude Coal Tar Oil	22 kg. 70 kg.	Caustic Soda (36° Bé.) <sup>10</sup>	9 kg.
	Formula	No. 11.	
Talloil—Distillate Coal Tar Oil		Caustic Potash (36° Be.) <sup>11</sup>	12.5 kg.
	FORMULA	No. 12.	
Woolfat Fatty Acid, Crude Coal Tar Oil	e 25 kg. 65 kg.	Caustic Potash (36° Bé)12	12 kg.
	FORMULA	No. 13.	
Olein Coal Tar Oil	20 kg. 65 kg.	Caustic Soda (36° Bé.) Methyl Hexalin <sup>13</sup>	9 kg. 5 kg.
	FORMULA	No. 14.	
Talloil, Crude Coal Tar Oil		Caustic Soda (24° Bé.) Alcohol, Denatured 14	15 kg. 3 kg.
	FORMULA	No. 15.	
Olein Colophony	5 kg. 15 kg.	Coal Tar Oil Caustic Soda (36° Bé.) <sup>15</sup>	70 kg. 9 kg.
8 Bennett, Chem. Formu	lary, Vol.	5, p. 76 (1941).	

<sup>9</sup> Ibid., p. 75 (1941).

<sup>10</sup> Ibid.

<sup>11</sup> Ibid.

<sup>12</sup> Bennett, Chem. Formulary, Vol. 5, p. 75 (1941).

<sup>13</sup> Ibid.

<sup>14</sup> Ibid.

<sup>15</sup> Ibid.

	FORMULA	No. 16.	
Talloil, Crude		Coal Tar Oil	65 kg.
Colophony		Caustic Soda (24° Bé.) <sup>16</sup>	15 kg.
	FORMULA	No. 17.	
Talloil, Crude		Coal Tar Oil	70 kg.
Woolfat Fatty Acid		Caustic Soda (24° Bé.) <sup>17</sup>	11.5 kg.
	FORMULA	No. 18.	
Talloil, Crude		Coal Tar Oil	70 kg.
Woolfat Fatty Acid		Caustic Soda (36° Bé.) <sup>18</sup>	8 kg.
	FORMULA	No. 19.	
Colophony		Coal Tar Oil	60 kg.
Crude Montan Wax		Caustic Potash (40° Bé.)18	12 kg.

# Preparation:

Use iron or enamelled agitated kettles which are steamheated, or fired from below, and that can be emptied at the bottom.

Heat 50% of the coal tar oils (or their blends) with the saponifiable materials (colophony, olein, talloil, montan wax, woolfat fatty acid, etc.) to about 80°C.

Thin the saponification lye with 50-100% of water in a separate kettle, low-degree-lyes being more diluted than high-degree lyes.

The thinned caustics are stirred into the hot oils. Boil. The saponification starts when the content of the kettle starts "rising."

Stir continuously, watch the heating in order to prevent the kettle boiling over.

As soon as the saponification is done, add the remainder of the coal tar oil, which is to be heated separately to 70°C. before.

<sup>16</sup> Bennett, Chem. Formulary, Vol. 5, p. 75 (1941).

<sup>17</sup> Ibid.

<sup>18</sup> Ibid.

<sup>19</sup> Ibid

When all is well mixed, boil for a second time, until the material is (nearly) clear. Too much water in the preparation makes it cloudy. In such a case boil further.

Test by cooling a sample, which should be clear and show the right, well-bound consistency. If the cooled sample is gelatinous, water should be added.<sup>20</sup>

FORMULA No. 20.

	FORMULA	No. 20.	
Coal Tar Oil	60 lb.	Ammonia (sp.g. 0.96)	15 lb.
(Carbolineum) Talloil	25 lb.	Water	10 lb.
Saponify at about 4 10 times the amount of		Should emulsify read	lily with
	FORMULA	No. 21.	
Talloil	300 kg.	Tar Oil (Middle Fraction)	1,200 kg.
Caustic Soda (20°Be.) 28	0-290 kg.		200 kg.
	FORMULA	No. 22.	
Nicotine Sulphate (40%) Bentonite Soybean Oil		Sodium Lauryl Sulphate Water <sup>28</sup>	½ oz. 100 gal.
	FORMULA	No. 23.	
Cottonseed Oil Copper Sulphate (10% solution)		Hydrated Lime Water <sup>24</sup>	6 lb. 100 gal.
	FORMULA	No. 24.	
Nicotine Soft Soap	¾ oz. ⅓ lb.	Water <sup>25</sup>	10 gal.

<sup>&</sup>lt;sup>20</sup> Bennett, Chem. Formulary, Vol. 5, p. 75 (1941).

<sup>21</sup> Ibid.

<sup>22</sup> Ibid.

<sup>23</sup> Ibid.

<sup>&</sup>lt;sup>24</sup> Bennett, Chem. Formulary, Vol. 5, pp. 75-76 (1941).

<sup>25</sup> Ibid.

### FORMULA No. 25.

Caustic Soda Napthenic Acids	1.3 lb. 10-12 lbs.	Mineral Oil	50-72 lbs.
Maphiethe Fields			

# Dilute and mix with water as needed.2

#### FORMULA No. 26.

#### FORMULA No. 27.

20 11

Copal Oil Water	Morpholine Oleic Acid		45 lb.
		 771	1.1

Add the oleic acid to the copal oil, using stirring. Then add the morpholine, still using high speed stirring. Add the water slowly and keep up stirring during addition of water.

This copal emulsion when sprayed on Japanese beetles will prevent then from flying and kill them slowly.27

	FORMULA	No. 28.
D1 411 1	2 15	Water 28

Phenothiazine Soap	3 lb. ½ lb.	Water <sup>28</sup>	100 gal
Comp	/-		

## FORMULA No. 29.

Derris (4% Rotenone)	3 lb.	Water 29	100 gal
Sodium Lauryl Sulphonate	3 oz.		

Spray twice ten days apart.

#### FORMULA No. 30.

Arachis Oil	2.00 1.	Ammonia	0.50 1
Oleic Acid	0.75 1.	Water	100.00 1

J. S. 2,000, 843.

<sup>&</sup>lt;sup>27</sup> Bennett, Chem. Formulary, Vol. 5, p. 101 (1941).

 <sup>&</sup>lt;sup>23</sup> Bennett, Chem. Formulary, Vol. 5, p. 76 (1941).
 <sup>29</sup> Bennett, Chem. Formulary, Vol. 5, p. 76 (1941).

The oil is mixed with the oleic acid. The ammonia is added to 2 1. of water. The oil mixture is added to the ammonia solution slowly with high speed agitation.30

#### FORMULA No. 31.

Cottonseed Oil	1 gal.	Potash Fish-Oil Soap	1 pt.
Paradichlorobenzene	2 lb.	Water to make	2 gal.

Dissolve 2 pounds of paradichlorobenzene crystals in 1 gallon of cottonseed oil. To do this, in cool weather, heat the oil to about 90°F., before adding the chemical. Stir the oil until the crystals entirely dissolve before proceeding further. Stir the resultant mixture into 1 pint of potash fish-oil soap and then stir in enough water to make 2 gallons. This stock emulsion may be stored for several days in a sealed drum. Each gallon of the stock emulsion should be diluted with 3 gallons of water immediately before making the treatment.31

#### FORMULA No. 32.

A 5% petroleum emulsion (made by adding 5.1 petroleum to 1 kg. laundry soap dissolved in 10 1. boiling water) is recommended against thrips.32

#### FORMULA No. 33.

Pyrethrum Extract	2.	Neutral Soap	4 lb.
Kerosene		Water <sup>33</sup>	94 gal.
	Eanasyr 4	No. 24	

#### FORMULA NO. 34.

White Mineral Oil	85 oz.	Triamylamine	3 oz.
(Vis. 80-85)		Monomylamine Oleate	10 oz.

One oz. of above mixture is diluted with 200-400 oz. of water.84

<sup>30</sup> Bennett, Chem. Formulary, Vol. 5, p. 102 (1941).

<sup>31</sup> Bennett, Chem. Formulary, Vol. 5, p. 90-91 (1941).
32 Bennett, Chem. Formulary, Vol. 5, p. 76 (1941).
33 Bennett, Chem. Formulary, Vol. 3, p. 119 (1936).
34 Bennett, Chem. Formulary, Vol. 5, p. 76 (1941).

Water

#### FORMULA No. 35.

Glyceryl Oleate Oil Soluble Nicotine Con	2 npound 5	White Mineral Oil to Water, to suit	93
	FORMULA	No. 36.	
Glyceryl Oleate Aluminum Oleate Nicotine	1.5 <b>2.0</b> 2.0	Naphthenic Acid White Mineral Oil 36 Water To suit	3.1 91.4
	FORMULA	No. 37.	
Gum Ghatti Cresylic Acid	2.4 lb. 0.18 lb.	White Oil (80 sec. Saybol at 100°F.)	t 62.4 lb.

The concentrated emulsion may be prepared by intimately mixing the ingredients in a colloid mill or by passing the mixture through a centrifugal pump or in any other suitable manner to give a concentrated emulsifiable composition which may readily be diluted to yield an emulsion suitable for spraying purposes.<sup>37</sup>

#### FORMULA No. 38.

Nicotine Sulphate (40%)	1 pt.	Wetting Agent	½ oz.
Bentonite	5 1b.	("Wetanol")	
Soybean Oil	1 qt.	Water	100 gal.

Mix well until emulsified 38

#### FORMULA No. 39.

Diglycol Oleate

2 lb. Pyrethrum Extract

(Mineral Oil or Kerosene) 50 lb.

35 lb. 1.4-Toluido Anthraquinone 0.02 lb.

Mixing the above together gives a concentrated spray base free from alkalies. The active principle of pyrethreum is thus unaffected. Burning due to alkali is also eliminated.

The above concentrate emulsifies readily on stirring in

<sup>\*5</sup> Adams, U. S. 2,258,833 (1941).

<sup>86</sup> Adams, U. S. 2,258,833 (1941).

<sup>&</sup>lt;sup>37</sup> Bennett, Chem. Formulary, Vol. 3, p. 119 (1936). <sup>38</sup> Bennett, Chem. Formulary, Vol. 5, p. 76 (1941).

water with a pump. It is "quick-breaking" when sprayed on the foliage.39

In any of the following formulae, which does not include water, such a formula is an emulsion base. These bases form emulsions on mixing with water.

### FORMULA No. 40.

Mineral oil 82.5, oleic acid 8.5, phenol 1.0, isoamyl alcohol 3.0, ethylene glycol monoethyl ether 1.5, caustic potash, 50% solution, 3.5. The formula for the emulsifier alone, which can be added to various insecticide liquids as required, contains; oleic acid 48.6, phenol 5.7, isoamyl alcohol 17.1, ethylene glycol monoethyl ether 8.6, caustic potash, 50% solution, 20. Amyl alcohol can be substituted for isoamyl alcohol, but the emulsifier is somewhat less stable; substitution of pine distillates requires special precautions. An emulsifier for miscible mixtures is oleic acid 32.5, cresol U.S.P. 32.5 "Yarmor" pine oil 10.0, caustic potash, 50% solution, 15.0 and kerosene 10.0. Fifteen percent of this emulsifier forms a miscible mixture which is stable on dilution with water. Phenol cannot be substituted for cresol in this formula. A combined spreader and emulsifier contains: oleic acid (high sapon. no.) 25.0, cresol U.S.P. 15.0, caustic potash, 50% solution, 20.0, kerosene 5.0. It has high emulsifying and spreading powers. In some instances a terpineol can be substituted for varmor pine oil, and London resin and pine tar oil (sp. gr. 1:035) for cresol. The formulas may be used with many but not all insecticide liquids; modifications are sometimes necessary.40

#### FORMULA No. 41.

Petroleum Oil (40-100 Sec. Cetyl Alcohol 41 0.2-10 1b. 100 lb. Saybolt)

<sup>39</sup> Ibid., Vol. 1, p. 215 (1933).

Bennett, Chem. Formulary, Vol. 5, pp. 76-77 (1941).
 Bennett, Chem. Formulary, Vol. 4, p. 105 (1939).

FORMULA No. 42.

Pyrethrum Extract, 15% 100 kg. Turkey Red Oil, Neutral 500 kg.

Apply in 0.5-1% aqueous dilution.42

FORMULA No. 43.

Pyrethrum Extract, 15% in acetone 100 kg.

Derris Extract in Acetone, (8% Rotenone) 100 kg.

Turkey Red Oil, Neutral 43 800 kg.

FORMULA No. 44.

Pyrethrum Extract (Kerosene)

1 gal. Carbon Tetrachloride 44 4 gal.

### FORMULA No. 45.

1 gram of pure rotenone is dissolved in 100 cc. pyridine. 1 cc. of this solution is added to 100 cc. distilled water and shaken gently. A pale opalescent colloidal solution results which shows no evidence of separation over an extended period of time.<sup>45</sup>

#### FORMULA No. 46.

5 grams of pure rotenone is dissolved in 100 cc. pyridine. 1 cc. of this solution is added to 100 cc. distilled water and shaken gently. A deeply opalescent colloidal solution results which shows no evidence of separation over an extended period of time. 46

### FORMULA No. 47.

1.2 grams of pure rotenone is dissolved in 100 cc. a-picoline. 1 cc. of this solution is added to 100 cc. distilled water and shaken gently. A colloidal solution of medium opalescence

<sup>42</sup> Ibid., p. 107 (1939).

<sup>43</sup> Ibid.

<sup>44</sup> Ibid., p. 104 (1939).

<sup>45</sup> Ibid., Vol. 2, p. 272 (1935).

<sup>46</sup> Bennett, Chem. Formulary, Vol. 2, p. 273 (1935).

results which shows no evidence of separation during a considerable period of time.<sup>47</sup>

FORMULA N	No. 4	8.
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Water	5 cc.	"Cellosolve"	10 cc.
Caustic Potash	7.4 g.	Oleic Acid	33.3 cc.
Pine Tar Oil	44.3 cc.		

Mix in the order given.4

### FORMULA No. 49.

Water	5.00	Ethylene Glycol Monoethyl	
Potassium Hydroxide		Ether	10.00
(92% Flaked)	7.40	Oleic Acid 49	33.30
Pine Tar Oil (Sp. Gr. 1.035)	44.30		

Mix in the order given.

FORMULA No. 50.

Soap 8 oz. Water 1 gal.

Boil and while mixing vigorously, add slowly

Pine Tar oil (d. 1.065) 50 6 oz.

### FORMULA No. 51.

Kerosene	90 g.	Triethanolamine	g
Oleic Acid	8 g.	Water 51	100 g.

This is made into a primary emulsion and may be diluted with one or two gallons of water for washing infested animals.

#### FORMULA No. 52.

Naphthalene	1 oz.	Cyclohexanol	2 oz.
Kerosene	4 oz.	Creosate 52	1 oz.

<sup>47</sup> Ibid.

<sup>48</sup> Ibid., Vol. 3, p. 118 (1936).

<sup>49</sup> Ibid., Vol. 4, p. 105 (1939).

<sup>50</sup> P. C.

<sup>&</sup>lt;sup>51</sup> Ibid., p. 115 (1939).

<sup>52</sup> Ibid., p. 104 (1939).

Dissolve by mixing and add slowly with vigorous stirring

Liquid Soap 32 oz. Water

120 oz.

FORMULA No. 53.

Phenol

1 lb. Water 58

3 gal.

Fish Oil Soap

1 lb.

Emulsify by stirring vigorously.

### Fruit Protective Emulsions

### FRUIT COATING WAX

### FORMULA No. 1.

Caustic Soda	6	Carnauba Wax	55
Triethanolamine	20	Shellac	100
Stearic Acid	42	Water	2000
Paraffin Wax	165		

### Dilute above before use.1

### FORMULA No. 2.

Paraffin Wax Carnauba Wax	553 68	Triethanolamine Water (Containing soda		98
Cottonseed Oil Oleic Acid	98 183	ash) <sup>2</sup>	То	suit

#### FORMULA No. 3.

Paraffin Wax	168.0	Sodium Bicarbonate	6.6
Beeswax	42.0	Salt	2.2
Oleic Acid	22.0	Water 2n	599.2

It is necessary that the soap be formed simultaneously with the first stage of emulsification by the addition of the alkali, in a minimum amount of water, to a mixture of the fatty acid and molten wax. These emulsions will readily coat fruits and vegetables with nonwaxy surfaces; but spreading agents

<sup>53</sup> Ibid., p. 109 (1939).

<sup>&</sup>lt;sup>1</sup> U. S. 2,153,487.

<sup>&</sup>lt;sup>2</sup> MacRill, U. S. 2,019,758 (1935).

<sup>&</sup>lt;sup>2a</sup> Trout, J. Coun. Sci. Ind. Research 15, 26 (1942).

are necessary when they are used for waxy fruits. Alkyl naphthalenesulfonates are suitable spreading agents and also inhibit breaking of the emulsions.

#### SHEEP DIPS

	Formul	a No. 1.	
Hydrocarbons Pyridin Cresylic Acid	46.78 g. 2.36 g. 21.67 g.	Rosin Soap Water <sup>3</sup>	5.39 g. 18.10 g. 5.70 g.
	Formul	a No. 2.	
Tar Oil Paraffin Oil Lanclin		Crude Cresylic Acid Soft Soap <sup>4</sup>	3.70 g. 21.00 g.

The tar oil used is that oil coming over between 180° to 220°C. and containing 20.25 per cent phenol. All the materials are mixed together in a boiler, and gradually heated until all the soap and lanolin have quite dissolved and then boiled gently for a short time, cooled and packaged, and is

T-	~ ~	•
FORMULA	No	. 1

Tar Oil	41.80 g.	Crude Cresylic Acid	3.70 g.
Paraffin Oil	33.50 g.	Soft Soap	21.00 g.

made in the same manner as the previous dip.5

<sup>&</sup>lt;sup>3</sup> Bennett, Chem. Formulary, Vol. 5, p. 112 (1941).

<sup>4</sup> Ibid., p. 113 (1941).

<sup>5</sup> Ibid.

# CHAPTER II

# CUTTING OILS, SOLUBLE OILS, MISCIBLE OILS

### FORMULA NO. 1.

Sodium Corn Oil Soap	14	Water White Rosin	10
Water	6	"Carbitol"	2
Mineral Oil	64	Diethylene Glycol	4

This oil is clear and will not become cloudy when cooled to a temperature of 60°F. It will not become covered with a film after standing exposed to the air at a temperature of 80°F. over a long period of time or at a temperature of 200°F. for one day. This oil will readily emulsify with water after standing exposed to the air at 200°F. for two days. Aqueous emulsions containing this oil are very stable even at a temperature of 200°F. In general, stable aqueous emulsions are prepared by using 1% to 35% of this oil, although stable aqueous emulsions can be prepared by using proportions of the oil outside these limits.¹

### FORMULA No. 2.

Kerosene	60 g.	Sulfonated Coconut Oil	20 g.
Sulfonated Castor Oil	20 g.	(Neutralized with Rosin	
Alcohol	1 g.	Soap)	
	- 0.	CF)	20 g.

This forms an emulsion when mixed with water.2

<sup>&</sup>lt;sup>1</sup> U. S., 1,965,935.

<sup>&</sup>lt;sup>2</sup> U. S., 2,021,448.

### FORMULA No. 3.

a. Oleic Acid	15	b. Kerosene	10
Rosin	15	c. Trigamine	20

Melt at 85°C. with high speed stirring. Add c and continue stirring. Then turn off the heat and add b slowly with stirring. This gives a clear transparent liquid which emulsifies readily in water.<sup>3</sup>

### FORMULA No. 4.

Mineral Oil		Glycostearin
Aerosol OT	(100%)	

Heat together to 100-120°C. cool to 80°C. and then add slowly with stirring to water (100 parts) (heated to 80°C.)<sup>4</sup>

### FORMULA NO. 5.

Rosin	15	Isopropyl Alcohol	10
Red Oil	15	"Wetanol" or other Wetting	
Mineral Oil	10	Out Agent	1/16
Potassium Hydroxide (45%)	5		

Warm together and mix until uniform.5

### FORMULA NO. 6.

a. Mineral Oil (Spindle Oil)	80	b. Caustic Potash (40° Bé)	6
Tall-Oil, Refined	20°	c. Methylhexalin 6	1–2

Saponify a with b, clear with c.

### FORMULA No. 7.

Mineral Oil	71–74	Caustic Potash	81/4-91/2
Castor Oil	81/4-91/2	Soda Ash	0.6-11/4
Rapeseed Oil	81/491/2		

Mix and dilute with water.7

<sup>&</sup>lt;sup>8</sup> Bennett, Chem. Formulary, Vol. 4, p. 207 (1939).

<sup>4</sup> P. C.

<sup>&</sup>lt;sup>5</sup> Bennett, Chem. Formulary, Vol. 4, p. 207 (1939).

<sup>6</sup> Ibid., Vol. 3, p. 210 (1936).

<sup>&</sup>lt;sup>7</sup> Bennett, Chem. Formulary, Vol. 3, p. 210 (1936).

	FORMULA	No. 8.	
Mineral Oil	75	Naphthenic Acid Sludge 8	25
	Formula	No. 9.	
Mineral Oil	75	Naphthenic Soap 9	25
	Formula	No. 10.	
Mineral Oil Oleic Acid	32 10	Alcohol Trigamine	

The above are thoroughly mixed cold and form a clear transparent oil which readily emulsifies on pouring into water with slight stirring.<sup>10</sup>

For	RMULA	No. 11.	
Light Mineral Oil Sulphonated Cod Oil Olein	65 <b>27</b> 10	Water Caustic Soda Cresol <sup>11</sup>	5 <b>2</b> 1
For	RMULA	No. 12.	
Alcohol, Denatured Diglycol Laurate	15 85	Mineral Oil 12	400
For	RMULA	No. 13.	
Pine Oil "Carbitol" "Sulfatate" (or other water- soluble sulphonic acid-esters	75 25	or salts) Water Diglycol Laurate Mineral Oil	20 <b>20</b> 360 1600
The "sulfatate" is disso	lved	in water and mixed	well into

The "sulfatate" is dissolved in water and mixed well into the other ingredients. This oil disperses in hard waters without scum formation.<sup>13</sup>

### FORMULA No. 13A.

Sulphonated Castor Oil	15	Diglycol Laurate	80
"Carbitol"	5	Mineral Oil 14	410

<sup>8</sup> Ibid., Vol. 4, p. 187 (1939).

<sup>9</sup> Ibid.

<sup>10</sup> Ibid.

<sup>11</sup> Bennett, Chem. Formulary, Vol. 4, p. 207 (1939).

<sup>12</sup> Ibid., p. 187 (1939).

<sup>18</sup> Ibid., p. 95 (1939).

<sup>14</sup> Ibid.

#### FORMULA No. 14.

(1) Sodium Abietate 10 (3) Mineral (2) Pine Oil 10	(Paramn) Oil 144	40
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Mix (1) and (2) and then add (3).

### FORMULA No. 15.

(1) Sodium Abietate	31	(3) Denatured Alcohol	4
(2) Rosin Oil	10	(4) Mineral (Paraffin) Oil 15	159

The above oils give rich creamy emulsions with water.

### FORMULA No. 16.

Oleic Acid Mineral Oil		Trigamine Alcohol <sup>16</sup>	<b>5</b> 3
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### FORMULA No. 17. .

Rosin	20	Diethylene Glycol	20
"Petronate"	100	Mineral Oil 17	840

### FORMULA No. 18.

Olein or Wool Grease	12.25	Mineral Oil	2.45
Animal or Vegetable Oil	7.50	Caustic Soda Solution 18	77.80

### FORMULA No. 19.

Paraffin Oil	48 fl. oz.	Red Oil	15 fl. oz.
Water	34 fl. oz.	Sodium Hydroxide 18a	1.8 oz.

The sodium hydroxide, carefully weighed, is dissolved in the water and the red oil and paraffin oil which have been previously mixed are run in while stirring rapidly. This forms a white milky preparation.

#### FORMULA No. 20.

Paraffin Oil (28° Bé.)	33	Sulphonated Red Oil (75%)	33
Sulfonated Castor Oil (75%)	33	Manipulation; Mix at 40°C.19	

<sup>14</sup>a Bennett, Chem. Formulary, Vol. 1, p. 428 (1933).

<sup>15</sup> Ibid.

<sup>&</sup>lt;sup>16</sup> Ibid., Vol. 4, p. 187 (1939).

<sup>&</sup>lt;sup>17</sup> L. Sonnenborn Sons, N. Y. C.

<sup>&</sup>lt;sup>18</sup> Erbs, Fr. 484,382 (1917).

<sup>18</sup>a P. C.

<sup>19</sup> P. C.

### FORMULA No. 21.

Paraffin Oil (28 to 30° Bé) Rosin Oleic Acid		Caustic Soda Water Alcohol <sup>20</sup>	3 10 7
	FORMULA	No. 22.	
Potash Soap Olein	30 7	Cyclohexanol Paraffin Oil <sup>21</sup>	2 32
	FORMULA	No. 23.	
Sodium Naphthenic Sulfo- nate Naphthenic Acid Sodium Hydroxide	12.00 3.00 0.50	*	0.50 2.00 0.05 81.95
	FORMULA	No. 24.	
Sodium Naphthenic Sulfo- nate Rosin Sodium Hydroxide	12.00 4.60 0.50		0.50 2.00 0.05 80.35
	FORMULA	No. 25.	
Pine Oil Red Oil Caustic Potash (50%)	2.5 12.0 4.	Alcohol, Anhydrous Potassium Oleo-Abietate <sup>23</sup>	5. 16.
	FORMULA	No. 26.	
Pine Oil	80	Double Sulphonated Castor Oil <sup>23a</sup>	20
SOLUBLE OIL STABLE TO SEA WATER			
	FORMULA	No. 27.	

Pine Oil	12	"Sulfatate" (Or other water-	
Red Oil	9	soluble sulfonated hydro-	
"Carbitol"	8	carbon) 24	6
Diglycol Laurate	65	,	•

<sup>&</sup>lt;sup>20</sup> Bennett, Chem. Formulary, Vol. 3, p. 210 (1936).

<sup>&</sup>lt;sup>21</sup> Ibid., Vol. 2, p. 303 (1935).

<sup>&</sup>lt;sup>22</sup> Zimmer, U. S. 2,230,556 (1941).

<sup>23</sup> Bennett, Chem. Formulary, Vol. 4, p. 96 (1939). 23a P. C.

<sup>&</sup>lt;sup>24</sup> Bennett, Chem. Formulary, Vol. 4, p. 95 (1939).

Cutting Oils,	Soluble	Oils. Mis	cible Oils	221
•	ORMULA	•		
Miscibol Oleic Acid Caustic Potash (45%)	16. 12. 3.	Isopropanol Pine Oil <sup>25</sup>	•	5. <b>2.</b> 5
I	ORMULA	No. 29.		
Sodium Abietate Oleic Acid	25 10	Pine Oil Isopropanol	26	4 6
F	ORMULA	No. 29A.		
Steam Distilled Pine Oil Sulphonated Castor Oil (75%	50 6) 50	Caustic Soda Water	ı (27° Bé.)	10 40
Heat the pine oil to sulphonated castor oil, with agitation, maintain constant agitation. Who add the water slowly, co rapidly. <sup>27</sup>	then ac ing the en a ne	dd the caus temperatur arly clear s	stic soda e noted al olution is	gradually oove with obtained,
I	FORMULA	No. 30.		
Soya Oil Fatty Acid or Linseed Oil Fatty Acid	25	Pine Oil		35
Warm gently with th	e follo	wing solution	on:	
Water Caustic Potash (50° Bé)	10 10	Pine Oil 28		160
I	ORMULA	No. 31.		
Spindle Oil, Refined 4-5 E. (20°C.) Oleic Acid, Distilled Sodium Hydroxide	50.0 20.0	Alcohol, To clear <sup>29</sup>	make	about 12.5
(29° Bé.) abou	ıt 12.5			

<sup>25</sup> P. C.

<sup>26</sup> P. C.

<sup>27</sup> Bennett.

 <sup>&</sup>lt;sup>28</sup> Ibid., Vol. 3, p. 325 (1936).
 <sup>29</sup> Bennett, Chem. Formulary, Vol. 4, p. 187 (1939).

### Practical Emulsions

### FORMULA No. 32.

Spindle Oil	100.0	Triethanolamine	0.5
Oleic Acid	12.0	Caustic Potash (38° Bé)	4.5
Butanol	7.0	•	

Heat the oils to about 70°C., add the butanol, and to this the triethanolamine. Add the lye, and leave standing hot to clarify.<sup>30</sup>

### FORMULA No. 33.

Talloil, Refined	20	Alcohol	To clear
Spindle Oil, Refined Caustic Soda (38° Bé.)	<b>72</b> 8	Fatty Acid 31	A little, to clear

### FORMULA No. 34.

Naphthenesulphonic Acids	15	Caustic Potash		
Olein (or Liquid Wool Fatty		(25° Bé.)	Until n	eutral
Acid)	5–7	Hexalin and Tetralin	(1:1)82	2 3-4
Spindle Oil, Refined (60°C.)	75	;	. ,	

### FORMULA No. 35.

Rosin Soap Red Oil	25 10	Pine Oil Alcohol, Anhydrous 33	4 6
	FORMULA	No. 36.	
Olein Spindle Oil Refined	16 73	Caustic Soda (36° Bé.) Alcohol, Denatured §4	6 5
	Formula	No. 37.	
Olein Spindle Oil, Refined	20 72	Caustic Potash 35 (40° Bé.)	8

### FORMULA No. 38.

Talloil, Refined Spindle Oil, Refined	20 70	Caustic Potash 36 (36° Bé.)	10
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<sup>30</sup> Ibid.

<sup>31</sup> Ibid., p. 207 (1939).

<sup>32</sup> Ibid., Vol. 3, p. 213 (1936).

<sup>38</sup> Bennett, Chem. Formulary, Vol. 4, p. 95 (1939).

<sup>34</sup> Ibid., p. 206 (1939).

<sup>35</sup> Ibid.

Cutting	Oils, S	oluble	Oils,	Miscible	Oils	223
	Fo	RMULA	No. 39			
Talloil, Refined Spindle Oil, Refined		20 72	Caustio	Soda 37 (	36° Bé.)	8
	Fo	RMULA	No. 40	•		
Train Oil Fatty Acid Spindle Oil, Refined		20 70	Caustic Hexali	Potash (4	40° Bé.)	7 3
	Fo	RMULA	No. 41			
Naphthenic or Hydr Acid Spindle Oil, Refined		12 80	Caustic Benzol	Soda (36 ine <sup>39</sup>	° Bé.)	6 2
	Fo	RMULA	No. 42	•		
Colophony (Rosin) Olein		8 6		e Oil, Refin Soda <sup>40</sup> (		78 8
	Fo	RMULA	No. 43			
Turkey Red Oil Olein		15 10		e Oil, Refin Potash 41		65 10
	Fo	RMULA	No. 44			
Bone Fat Fatty Acid	I	20 5		e Oil, Refin Soda 42 (		63 12
	Fo	RMULA	No. 44a	ı,		
Lanolin Fatty Acids Spindle Oil, Refined		16 75	Causti	e Potash 48	(24° Bé.)	9
Formula No. 44b.						
Woolfat Fatty Acids		7 8		e Oil, Refin Soda 44 (?		77 8
37 Ibid. 38 Bennett, Chem. 39 Ibid. 40 Ibid. 41 Ibid. 42 Ibid. 43 Bennett, Chem. 44 Ibid.						

### FORMULA No. 45.

Olein	Caustic Potash (40° Bé.)	7
Spindle Oil, Refined	Water <sup>45</sup>	30

The amount of caustic must be varied since the saponifiable materials are not constant in acidity.

FORMULA NO.	. <del>4</del> 6.
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Warm and mix vigo	rously.46		
	FORMULA	No. 47.	
Sulphonated Oil Soap Cottonseed Oil		Oleic Acid Mineral Oil Water <sup>47</sup>	10.5 2.0 50.0
	FORMULA	No. 48.	
"Carbitol" Diglycol Laurate	5 95	Orthodichlorbenzol 48	400
	FORMULA	No. 49.	
Sulfonated Castor Oil Turpentine	70 10	Tetrahydronapthalene Pine Needle Oil 49	15 5
	FORMULA	No. 50.	
Tetralin Turpentine	15 10	Pine Needle Oil Sulfonated Vegetable Oil 50	70
	FORMULA	No. 51.	
Sal Soda Lard Oil	⅓ lb. ½ pt.		½ pt. 10 qt.
Boil the above ingre	dients fo	or one-half hour and place	ce in

suitable containers for use as needed.49

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45 Ibid.
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<sup>46</sup> Ibid., p. 207 (1939).

<sup>47</sup> Bennett, Chem. Formulary, Vol. 4, p. 207 (1939).

<sup>48</sup> Ibid., Vol. 4, p. 95 (1939).

<sup>49</sup> Muncheburg, Fr. 674,816 (1929). 50 Muncheburg, Fr. 674,816 (1929).

<sup>49</sup> Bennett, Chem. Formulary, Vol. 2, p. 303 (1935).

### METAL DRAWING COMPOUNDS

	FORMUI	A No. 1.	
Lard Oil, Prime Diglycol Laurate	40 lb. 10 lb.	Water 50	50 gal.
	Formu	A No. 2.	
<ol> <li>Soap, Neutral</li> <li>Diglycol Laurate</li> <li>Lard Oil, Prime</li> </ol>	10 lb. 7 lb. 13 lb.	4. Mineral Oil 5. Water	20 lb. 200 gal.

Warm, together, 1, 2, 3 and 4 until clear and pour into water slowly and mix well. In certain cases it is desirable to add

White Lead 51	3 1b.		
	FORMUL	a No. 3.	
Diglycol Stearate Glycowax A	6 2	Water 52	56
	FORMUL	A No. 4.	
Sodium Alginate Tallow	1 4	Soap Water <sup>53</sup>	2 195
	FORMULA	A No. 5.	
Tallow Rape Seed Oil	3.0 1.5	Soap Water	0.5 95.0

Warm together and emulsify. In using maintain pH at 8-9.54

### FORMULA No. 6.

One part of soluble alginate, four parts of tallow, two parts of soap, and one hundred and ninety five parts of water.<sup>55</sup>

<sup>&</sup>lt;sup>50</sup> Ibid., Vol. 4, p. 202 (1939).

<sup>51</sup> Ibid.

<sup>52</sup> Ibid.

<sup>&</sup>lt;sup>53</sup> Bennett, Chem. Formulary, Vol. 3, p. 211 (1936).

<sup>54</sup> Ibid., Vol. 4, p. 202 (1939).
55 Ibid., Vol. 2, p. 300 (1935).

### FORMULA No. 7.

Tallow Yellow Soap	60 lb. 15 lb.	W	ater	•					92	gal.
Heat and stir until s	mooth	56								
	Formu	LA N	Io. 8							
Sulfated Higher Fatty Alco Tallow	ohol 3		/ater	- 57						20
NAPHT	HENIC	MI	SCII	3LE	OII	LS				
Formula 1. Potassium naphthenate from—	No. 1	2	3	4	5	6	7	8	9	10
Heavy gas oil Light lube oil Heavy lube oil		48	37	37	37	23	20	30	13	30
<ul><li>2. Light lube oil</li><li>3. Common solvents</li></ul>	20	35	50	45	50	58	62	55	69	64
Group 1—Water soluble Ethylene Glycol Diethylene Glycol Group 2—Oil soluble		5	5	5	5	5	5	5	5	3
Sub-group A Pine Oil Alpha Terpineol Octyl Alcohol		10	5	10	5	5	5	10	5	
Sub-group B Rosin Cresylic Acid		;				5	5		5	3
4. Water 58		2	3	3	3	4	3		3	

<sup>&</sup>lt;sup>56</sup> Ibid., Vol. 3, p. 209 (1939).
<sup>57</sup> Williams, U. S. 2,152,396 (1939).
<sup>58</sup> Bradley, U. S. 2,289,536 (1942).

### CHAPTER III

### EMULSIFYING AGENTS

### FORMULA NO. 1.

10 kilograms of casein and 10 kilograms of citric acid are stirred up and heated with 100 liters of water, and the solution is then diluted with a further 200 liters of water, the undissolved parts being removed. Potash lye is then added hot until the concentration is pH 7.4, and the solution is carefully evaporated. A homogeneous residue is thus obtained which is completely soluble in water, and is admirably adapted either as an emulsifying agent or detergent, either by itself or with fat solvents or neutral soap, especially for wool scouring.<sup>1</sup>

#### FORMULA No. 2.

a.	Pectin, Dry	4-5		Gelatin	1/2
	Agar-Agar	1	b.	Water, Boiling	100

Dissolve a in b, cool. The smooth jelly that forms is liquefied once again by stirring and run through a homogenizer.

The viscous liquid thus formed can be used to emulsify oils in varying amounts.<sup>2</sup>

<sup>2</sup> Ibid., Vol. 4, p. 95 (1939).

<sup>&</sup>lt;sup>1</sup> Bennett, Chem. Formulary, Vol. 2, p. 181 (1935).

### FORMULA No. 3.

Cottonseed Oil	<i>7</i> 5	Potassium	Carbonate
Diethylene Glycol	22		

Heat with good stirring, for 1 hour, at 125-130°C.

This is soluble in mineral oil and disperses in warm water.3

### FORMULA No. 4.

Castor Oil	46.6	Diethylene Glycol	44.0
Soya Bean Oil	112.5	Potassium Carbonate	2.0

Method as above.4

### FORMULA No. 5.

Coconut Oil	137	Potassium Carbonate	2
Diethylene Glycol	44		

Method as above.5

### FORMULA No. 6.

Palm Kernel Oil	150	Potassium Carbonate	5
Diethylene Glycol	44		

Method as above.6

#### FORMULA No. 7.

Castor Oil, Blown	85.0	Sodium	Hydroxide 7	(10%)	6.7
Castor Oil Fatty Acids	15.0		-	. , ,	

FORMULA No. 8.

Diglycol Laurate 80 "Carbitol" 20

A good white water in oil cream emulsion is obtained from

Above emulsifier	15	Water 8	45
		// utuz	-10
Mineral Oil	40		

<sup>&</sup>lt;sup>3</sup> Bennett, Chem. Formulary, Vol. 4, p. 99 (1939).

<sup>4</sup> Ibid.

<sup>5</sup> Ibid.

<sup>6</sup> Ibid.

<sup>&</sup>lt;sup>7</sup> Bennett, Chem. Formulary, Vol. 4, p. 99 (1939).

<sup>8</sup> Ibid.

1	Emulsifyin	g Agents	229
	Formula	No. 9.	
Pectin Agar	4–5 1	Gelatin Water (Boiling) <sup>9</sup>	100 cc.
	Formula	No. 10.	
Soya Bean Flour Water	1.0 150.0	Caustic Potash	0.1
Heat together and	stir until 1	miform.io	
	Formula	No. 11.	
Pectin Agar		Gelatin Gum Arabic <sup>12</sup>	0-1 0-5
	FORMULA	No. 12.	
Gum Tragacanth Gum Arabic	20 5	Sodium Bicarbonate 13	13
	FORMULA	No. 13.	
Gum Tragacanth Locust Bean Gum	20 15	Sodium Bicarbonate 14	13
	Formula	No. 14.	
Powdered Acacia Powdered Tragacanth Sugar	5 5 5	Starch Boric Acid	5 1

Mix intimately. Use 1 part of above powder to every 32 parts of emulsion to be made. Eight ounces of the oil to be emulsified is put into a dry 32 ounce bottle, and shaken with  $\frac{1}{2}$  ounce of the above. When the powder is evenly suspended, 8 ounces of water is added at once, and the mixture is well shaken until a perfect emulsion is formed.<sup>15</sup>

### FORMULA No. 15.

Aerosol OT (100%)	40	Triethanolamine	18
Oleic Acid	42		

<sup>9</sup> Ibid.

<sup>10</sup> Ibid.

<sup>12</sup> Preiser, Brit. 486,463 (1938).

<sup>13</sup> P. C.

<sup>14</sup> P. C.

<sup>15</sup> Bennett, Chem. Formulary, Vol. 2, p. 181 (1935).

The Aerosol OT (100%) is dissolved in the oleic acid by heating to 50°C to 60°C. The mixture is cooled to room temperature and the triethanolamine added. 5% of this emulsifier can be mixed with 95% of oil and to it added 30% to 50% water, whereupon a thick creamy paste will be obtained, which can be further diluted with additional water.<sup>16</sup>

#### FORMULA NO. 16.

55 parts of lauryl amide are mixed with 50 parts of formaldehyde (30 per cent), 0.5 part of potassium hydroxide solution (50 per cent concentration) and 300 parts of water. The mixture is agitated for half an hour at a temperature of 60 degrees C. It is agitated for another hour after the addition of 50 parts of phenol and 10 parts of concentrated solution of hydrochloric acid. The viscous oil, obtained from this treatment, is an excellent emulsifier. It is mixed with 200 parts of sulfuric acid (monohydrate) at a temperature of 60° to 70°C., and after the sulfonated mixture has been agitated for several hours, it is mixed with 200 parts of ice and slowly heated with agitation to a temperature of approximately 70°C. After agitation is stopped, the sulfonate formed quickly separates in the state of a viscous oil over an aqueous solution of sulfuric acid. The latter is removed and the oily sulfonic acid is dissolved in water, neutralized and evaporated. A product is finally obtained which possesses high detergent powers both in hard and soft water and is resistant to the action of hot acids and alkalies

### FORMULA No. 17.

380 kilograms of octadecanolamine hydrochloride are introduced into 200 kilograms of trichlorethylene and sulphonated at 10°C. with 120 kilograms of chlorsulphonic acid. The product is pressed on ice, washed with saturated

<sup>&</sup>lt;sup>16</sup> Amer. Cyanamid & Chem. Corp., New York.

Glauber's salt solution in order to remove the excess of sulphuric acid and acetic acid and neutralized with caustic soda solution.<sup>17</sup>

<sup>&</sup>lt;sup>17</sup> Bennett, Chem. Formulary, Vol. 2, p. 183 (1935).

### CHAPTER IV

## BITUMINOUS EMULSIONS

### ASPHALT EMULSIONS

### FORMULA NO. 1.

Asphalt	64.05	Corn Gluten (or Soy Bean	
Water	35.80	Meal)	.64
Caustic Soda	83.	Green Acid Soap (Dry Basis)	0.23

The asphalt for use in this formula may be produced from Mid-Continent petroleum; it may have a melting point of about 110°F. and a penetration of about 130 at 77°F.

Green acid soap is a composition of preferentially water soluble petroleum sulfonic salts. This soap may be prepared by the neutralization of green acids, which are well known in the petroleum industry. If the green acid soap contains appreciable amounts of oil, a harder asphalt should be emulsified, to produce a residue of given penetration, than is necessary when the green acid soap is free from oil.

In preparing the emulsion the procedure is as follows: The caustic, farinaceous emulsifier, and green acid soap are mixed with water and heated to a temperature of about 200°F. This hot solution or mixture is placed in a suitable stirrer, agitator or mixer and is beaten by paddles, circulated by centrifugal pumps or dispersed between suitable rotors moving at high velocities.

The melted asphalt at about the same temperature is

slowly stirred into the solution and further agitated until complete emulsification has taken place. When high melting point asphalts are used it may be necessary to increase the temperature at which the asphalt is added, but it is desirable to keep the temperature of the emulsion below the boiling point of water so that the foaming due to the production of steam may be prevented.1

### FORMULA NO. 2.

A sodium oleate solution is made up to a concentration of 20 per cent by the addition of oleic acid and caustic soda to water at 90°C. This is then diluted with 9 times its volume of water heated to 90°C. The 2 per cent soap solution is run through a colloid mill with an equal amount of asphalt heated to not exceeding 100°C. The resultant emulsion contains equal parts of asphalt and water with 1 per cent by weight of soap.2

#### FORMULA NO. 3.

A hot dilute aqueous solution of alkali such as a solution of caustic soda of about 0.5% strength is prepared and about an equal weight of asphalt is melted; part of the melted asphalt is stirred into the hot solution slowly until scum begins to form on the surface, then a small quantity (suitably about 0.5% of the final product) of oleic acid is added, followed by addition of the rest of the asphalt while the temperature and agitation are maintained and a small proportion of clav is added to give desired stability and adhesiveness.3

FORMULA NO. 4.

Rosin

100 Slaked Lime

3 P. C.

Bennett, Chem. Formulary, Vol. 2, p. 187-188 (1935).
 Bennett, Chem. Formulary, Vol. 1, p. 163 (1933).

Heat at 140°C. and mix until uniform. Add while mixing

Fuel Oil

103

Take 4 lbs. of above mixture and add to 100 lb. melted bitumen or asphalt and then disperse mixture in 0.05 N potassium rosinate solution to give a 57% bitumen content.<sup>4</sup>

### FORMULA No. 5.

A part of molten asphalt is added to a hot, dilute, aqueous solution of caustic soda, an emulsifying agent, e.g., oleic acid, is added, and then the remainder of the asphalt, all with agitation; at an appropriate time of the operation, e.g., before adding the asphalt, a small proportion of colloidal clay is added as a stabilizer. A suitable emulsion may contain asphalt 48–52%, water 46–48%, oleic acid 1%, sodium hydroxide less than 1%, bentonite 1%.

### FORMULA No. 6.

This consists in dissolving substantially 9 parts, by weight, of soap in substantially 78 parts of weight of warm water. To the soapy water thus formed is slowly added, with vigorous agitation, about 20 parts, by weight; of a low grade fuel oil or a crude oil having an asphaltic base, to produce a dispersion of the oil, in the warm soapy water.<sup>6</sup>

### FORMULA No. 7.

Asphalt Water Bentonite	500	Quebracho Soda Ash	30 10
Bentonite	30		

Combine the bentonite, quebracho, soda ash and water and heat to 200°F. While stirring, add the asphalt which

6 Ibid

<sup>&</sup>lt;sup>4</sup> Bennett, Chem. Formulary, Vol. 4, p. 97 (1939).

<sup>&</sup>lt;sup>5</sup> Ibid., Vol. 2, p. 186 (1934).

has been heated to approximately 200°F. Continue stirring until the asphalt is dispersed.<sup>7</sup>

FORMULA NO. 8.

Asphalt	Rosin Soap (50%)	118
Water	Pine Oil	40

Add the rosin soap to water and heat to 200°F. Heat the asphalt to 200°F, and add the pine oil. While agitating, slowly pour the asphalt into water and continue agitating until a smooth emulsion is formed.8

FORMULA No. 9.

Hydrous Magnesium	Water	20 gal.
Silicate	8–10 1b.	

Mix well and heat to a boil; then mix in an emulsifying machine with

Asphalt (Melted) 36 gal. Water (Boiling) 9 10 gal. Acetic Acid (0.1 N.) 250-420 cc.

### FORMULA NO. 10.

Shale asphalt, consisting of residues obtained after distilling crude shale oil, is heated to about 300° and distd. in steam at 325° to drive off traces of oil, and mixed with montan wax and a fatty acid at 120–30° in the proportions asphalt 50, wax 1–2 and fatty acid 0.5–2.5. The molten mixture is led in fine stream with stirring into an aqueous caustic soda of soap solution at 90°C. in proportions water 15–20, a caustic soda 0.3–0.6 and fatty acid 0.4–0.5. The emulsion is then boiled for 2–10 minutes, and may be mixed at a temperature not above 55°C. with rubber latex, such as Revertex.<sup>10</sup>

<sup>&</sup>lt;sup>7</sup> Bennett, Chem. Formulary, Vol. 1, p. 155 (1933).

<sup>8</sup> Ibid.9 Ibid., Vol. 4, p. 96 (1939).

<sup>10</sup> Ibid., p. 97 (1939).

#### RITUMEN EMULSIONS

### FORMULA No. 1.

1,000 g. bitumen (preferably asphalt) are heated to 90°C. 60 g. Swedish resin at 80–90°C. is then added and finally a solution of 20 g. caustic potash in 120 g. water, cooled to 60–70°C., is added. Vigorous stirring is needed throughout. 1000 g. hot water is poured in and when soap formation is ended a further 800 g. water is added.

### FORMULA No. 2.

Add 15 parts by weight of oleic acid to 250 parts by weight of asphalt flux oil, heating the mix to about 100°C., adding the same to 750 parts of water to which have been added 34 parts of aqueous ammonia whereby an emulsion of the asphalt flux oil, which has a viscosity substantially greater than that of water is produced, thereafter adding 1500 parts by weight of coal tar, which has a specific gravity of about 1.18 or more, heated to a temperature of about 70°C. to which has been added 45 parts of oleic acid. Vigorously agitate the tar with the emulsion of asphalt flux oil, and subject the resulting mixed emulsion to intensive mechanical disintegration, as for example, by passage through a colloid mill.<sup>2</sup>

### FORMULA No. 3.

An emulsion for road making contains Spramex bitumen 48, water 49.5, oleic acid 2 and calcined sodium carbonate 0.5%. The bitumen is warmed at 95–98°, and the oleic acid added. The water is heated separately with the sodium carbonate and the two liquids are introduced into the emulsifier. Another emulsion contains Spramex bitumen 50, mineral oil 2–2.5, resin soap 1.5–2, caustic potash 1, and water 45%. The bitumen is melted and the mineral oil added during

<sup>2</sup> Ibid.

<sup>&</sup>lt;sup>1</sup> Bennett, Chem. Formulary, Vol. 4, p. 97 (1939).

agitation. The water is heated to boiling, and in it are dissolved the soap and the caustic potash. The liquids are mixed at 95°. With more bitumen there must be added 1–2% of glue, starch, gelatin or sodium silicate, during or after emulsification.<sup>3</sup>

### FORMULA No. 4.

Melt 100 parts of bitumen, having a softening point of 45° to 50°C., and add 10.8 parts of rosin, and heat to 100–125°C. Then add 20 parts of kaolin (swollen in equal parts by weight with water), and 1.2 parts of sodium hydroxide, preheated to 70° to 80°C. Raise the temperature of this mixture to 100°C., and dilute with water to produce an emulsion containing 20 to 25% of solids.<sup>4</sup>

•		-	
	FORMULA	No. 5.	
red			1 lb. 50 lb.
PI	тсн ем	MULSION	
ch	180	Stearin Pitch	20
ether and ad	d to foll	owing at 100°C.	
ution	45 <b>35</b>	Caustic Potash	1
đ			
		Water, Boiling	120
*CA	SEIN SC	DLUTION =	
sh (50%)			494 11
	red PI The character and addition  d	FORMULA 35 lb. red 15 lb. PITCH EM 180 rether and add to following ation 45 35 d *CASEIN SO	PITCH EMULSION  180 Stearin Pitch  rether and add to following at 100°C.  150 Caustic Potash  35  d  Water, Boiling  *CASEIN SOLUTION =  56 Water

<sup>&</sup>lt;sup>3</sup> Bennett, Chem. Formulary, Vol. 1, p. 329 (1933).

<sup>4</sup> Ibid., Vol. 5, p. 72 (1941).

<sup>5</sup> Ibid.

<sup>&</sup>lt;sup>6</sup> Bennett, Chem. Formulary, Vol. 4, p. 97 (1939).

### TAR ASPHALT EMULSION

Shale Tar	38.15 lb.	Rosin	1.15 lb
Water	49.60 lb.	Potato Starch	0.76 lb
Mexican Asphalt	7.63 lb.	Anthracene Oil 7	1.34 lb
Casein	1.15 lb.		

<sup>&</sup>lt;sup>7</sup> Ibid., Vol. 5, p. 73 (1941).

### CHAPTER V

### CLEANERS AND SOAPS

### DRY CLEANERS' SOAPS

### FORMULA No. 1.

Xylol	10	Diglycol Laurate	7
Carbon Tetrachloride	10	Water	23

Add other materials slowly to water while mixing with a high speed stirrer.<sup>1</sup>

### FORMULA No. 2.

a. Oleic Acid	46.0	c. Alcohol	45.4
b. Caustic Soda (25%)	27.0	d. Trichlorethylene	900.0

Saponify a with b on a waterbath. Add c and reflux to clear solution. Add this clear alcoholic soap solution to d with stirring, and let cool.<sup>2</sup> Water emulsifies this fluid readily.

#### FORMULA No. 3.

Oleic Acid	370	Water	70
Stearic Acid	80	Benzine	395
Potassium Carbonate	80	Stronger Ammonia Water	5

Melt the stearic acid and dissolve it in the warmed oleic acid. To this add the warm benzine and mix thoroughly.

<sup>&</sup>lt;sup>1</sup> Bennett, Chem. Formulary, Vol. 4, p. 497 (1939).

<sup>&</sup>lt;sup>2</sup> Ibid.

Dissolve the carbonate in the water and add this, with constant stirring, into the benzine mixture. Finally add the ammonia and beat into a homogeneous paste.<sup>3</sup>

FORMULA No. 4.

Liquid Soap (15%) Hydrocarbon Solvent Turkey Red Oil or Other Emulsifier	40 10 25	Alcohol (Methyl Glycol, Ethanol, etc.) <sup>1</sup>	25
Fe	ORM UL.	No. 5.	
Trihydroxyethylamine     Stearate     Kerosene	5 16	<ul><li>3. Cresylic Acid</li><li>4. Water (Boiling)</li></ul>	1 45
Heat (1) and (2) unstirring with high speed r		solved; add (4) slowly then add (3).2	while
PIN	KE OI	L SOAP	
Fo	RMUL	No. 1.	
Glycol Laurate	5	Pine Oil	25
Mix and add to follow	ing w	hile stirring vigorously	
Water	50	Caustic Soda 3	1/4
Fo	RMUL	A No. 2.	
Water	8.0	Pine Oil	18.0

Mix the ingredients, while stirring in the order given, at a temperature of about 40°C.; finally adjust with red oil or alkali until a sample dissolved in alcohol is neutral to phenolphthalein.<sup>4</sup>

Water

Red Oil (Oleic acid)

17.5

44.0

2.5

10.0

Solid Caustic Soda

Alcohol

<sup>&</sup>lt;sup>3</sup> Ibid., Vol. 1, p. 76 (1933).

<sup>&</sup>lt;sup>1</sup> Bennett, Chem. Formulary, Vol. 4, p. 496 (1939).

<sup>&</sup>lt;sup>2</sup> Ibid., Vol. 1, p. 76 (1933). <sup>3</sup> Ibid., Vol. 3, p. 325 (1936).

<sup>\*</sup>Bennett, Chem. Formulary, Vol. 1, p. 84 (1933).

Cle	aners a	nd Soaps	241
	FORMULA	No. 3.	
Potash Corn Oil Soap	96–97	Pine Oil 5	4-3
EMITTO	ON COI	LVENT SOAPS	. 0
EMOLSI	.014 301	LVENI SUAPS	
	FORMULA	No. 1.	
Pine Oil Soya Bean Oil Fatty Acid	300 100	Water	60
Warm gently to be lie	quefied,	then add	
Caustic Potash (50° Bé.)	40		
1 part of the above mix	ces with	1	
Turpentine	4		
	O	r	
Benzoline	4		
	O	r	
Carbon Tetrachloride	4		
	O	r	
Dichloroethylene	4		
	<b>O</b> 1	r	
Naphtha	4		
These give excellent	emulsio	ons in water (1:1 to 1:2)	).
	Formula	No. 2.	
Pine Oil Soap (Above) Pine Oil	12.5 12.5		75
Vields a clear oil that o	duec ev	cellent emulsions with was	ter 1

Yields a clear oil that gives excellent emulsions with water.1

### FORMULA No. 3.

Oleic Acid	107.0	Triethanolamine	19.7
Butyl Cellosolve	27.0	Potassium Hydroxide	8.3
Cleaner's Naphtha	25.0	Water	13.5

<sup>&</sup>lt;sup>5</sup> P. C. <sup>1</sup> Bennett, Chem. Formulary, Vol. 3, p. 324 (1936).

The oleic acid, butyl cellosolve and naphtha are thoroughly mixed and heated to 140°F, in the absence of flames. In a separate container the potassium hydroxide is dissolved in the water and mixed with the triethanolamine. The water solution is then stirred into the oleic acid solution, and stirring is continued for about 30 minutes until a clear stable solution is produced.2

Fo	RMULA	No. 4.			
Linseed Oil Hexalin 256	500 0-300	Potash Lye Water <sup>3</sup>	(50°	Bé.)	199 1208
Fo	RMULA	No. 5.			
Linseed Oil Fatty Acids Methyl Hexalin	500 750	Potash Lye Water 4	(50°	Bé.)	208 292
Fo	RMULA	No. 6.			
Coconut Oil Fatty Acids Hexalin-Methyl Hexalin (1:1)	500 250	Potash Lye Water <sup>5</sup>	(50°	Bé.)	270 1300-1800

The ingredients are stirred together in a double boiler until a clear solution is formed; this is tested for alkalinity.

	FORMULA	No. 7.	
Linseed Oil Hexalin Potash Lye (50° Bé.)	184 275 73.5	Water Carbon Tetrachloride <sup>6</sup>	38 <b>7</b> 8 <b>0</b>
	FORMULA	No. 8.	
Coconut Oil Linseed Oil Hexalin	51 42 130	Potash Lye (50° Bé.) Water Carbon Tetrachloride 7	42 615 120

Equal weights of benzine or high-boiling petroleum distillates may be used in place of carbon tetrachloride.

<sup>&</sup>lt;sup>2</sup> Ibid., Vol. 1, p. 79 (1933).

<sup>&</sup>lt;sup>8</sup> Bennett, Chem. Formulary, Vol. 3, p. 322 (1936).

<sup>4</sup> Ibid.

<sup>5</sup> Ibid.

<sup>6</sup> Ibid.

<sup>&</sup>lt;sup>7</sup> Bennett, Chem. Formulary, Vol. 3, p. 322 (1939).

Cl	eaners a	nd Soaps	243
	FORMULA	No. 9.	
Soap Cyclohexanol	35 10	Water 8	55
	FORMULA	No. 10.	
Soap Trichloroethylene	28 10	Water Potassium Carbonate 9	60 2
	FORMULA	No. 11.	
Soap Trichloroethylene	30 25	Water 10	45
	FORMULA	No. 12.	
Soap Ammonia (0.880)	5 25	Cyclohexanol Water <sup>11</sup>	10 <b>60</b>
	FORMULA	No. 13.	
Soap Ammonia (0.880)	10 5	Tetralin Water <sup>12</sup>	10 75
AUTO	RADIAT	OR CLEANER	
Kerosene Orthodichlorobenzene Borax	25 25 5	Diglycol Oleate Water	5 40

The borax is dissolved in the water and the other ingredients are dissolved in the kerosene, and the two solutions are then emulsified together, making a stable emulsion.

When the cleaning composition is to be used, it is added to the water in the cooling system of an engine, and the engine is set in operation so that the water, by its circulation, carries the emulsion to all parts of the system. While the water is still cool the oily ingredients, together with the diglycol oleate dissolved in them, penetrate the solid deposits in the system,

<sup>8</sup> Ibid.

<sup>9</sup> Thid.

<sup>10</sup> Thid.

<sup>11</sup> Bennett, Chem. Formulary, Vol. 3, p. 322 (1939).

<sup>12</sup> Ibid.

and dissolve or soften any grease contained in the deposits. When the water is sufficiently heated, by the continued operation of the engine, the borax reacts with the diglycol oleate to produce a water-soluble soap, thus disintegrating the deposits and producing an oil-in-water emulsion. The cooling water and the emulsified materials are then drained from the system, and any remaining solids, having been loosened and disintegrated, may be flushed from the system with additional water.<sup>1</sup>

#### CHROMIUM PLATE CLEANERS

		· ·	
	FORMULA	No. 1.	
Amorphous Silica Bentonite Orthodichlorobenzene	5.0 1.0 10.0	Triethanolamine Oleate Oleic Acid Water <sup>2</sup>	3.0 0.5 80.0
	FORMUL	A No. 2.	
Bentonite Stearic Acid	5. 5.	Orthodichlorobenzene Water <sup>3</sup>	40. 50.
	FORMULA	No. 3.	
Mineral Oil Pine Oil Ammonium Oleate Ammonium Oxalate	13.0 3.0 2.0 3.0	Tripoli Ammonia Water <sup>4</sup>	25.0 0.5 54.0
G	RAPHITE	CLEANER	
Hexalin Pine Oil	2 qt. 4 qt.	Soap Solution (1%)	3 gal.

These are mixed together to form a fairly stable emulsion. Hot water is added while stirring constantly until the emulsion is diluted to 50 gallons.<sup>5</sup>

<sup>&</sup>lt;sup>1</sup> Bennett, Chem. Formulary, Vol. 5, p. 574 (1941). <sup>2</sup> Ibid.

<sup>3</sup> Ibid.

<sup>&</sup>lt;sup>4</sup> Bennett, Chem. Formulary, Vol. 5, p. 574 (1941).

<sup>&</sup>lt;sup>5</sup> Ibid., Vol. 4, p. 501 (1939).

#### INK CLEANER

### FORMULA No. 1.

a.	Naphtha	10	b. Ammonium Sulphate	25
	Trichlorethylene	20	Water	100
	Methyl Hexalin	5	c. Pumice Powder 000)	1
	Soft Soap	25	Chalk Powder	equal parts

Mix solution a with a paste prepared from b and c.1

### FORMULA No. 2.

a.	Pumice Powder	50	b. Turpentine	1
	Soap Powder	30	Methyl Hexalin	equal
	Trisodium Phosphate	15	Carbon Tetra-	parts
	Ammonium Sulphate	5	chloride <sup>2</sup>	]

### LACQUER REMOVER FOR HANDS

Butyl Acetate	13	Diatromaceous Earth	14
Soap	15	Sand	19
Carbon Tetrachloride	7	Water 3	25
Glycerin	7		

### LEATHER CLEANER

Castile Soap (Powd.)	6 Water	160
Castile Soap (1 owd.)	U Water	100

### Boil until dissolved; cool and add

Ammonium Hydroxide	6	Ethylene Dichloride 4	7
Glycerin	14		

#### GREASY LEATHER CLEANER

Water	10 gal.	Methyl Acetone	.5 gal.
Castile Soap	.75 lb.	Lemon Grass Oil 5	.15 lb.
Trichlorethylene Soan	3.5 lb.		

Dissolve soap in water by heating and stirring. Cool and stir in other ingredients.<sup>5</sup>

<sup>&</sup>lt;sup>1</sup> Bennett, Chem. Formulary, Vol. 5, p. 574 (1941).

<sup>2</sup> Thid

<sup>&</sup>lt;sup>8</sup> Bennett, Chem. Formulary, Vol. 4, p. 511 (1939).

<sup>&</sup>lt;sup>4</sup> Ibid., Vol. 1, p. 77 (1933). <sup>5</sup> Ibid., Vol. 4, p. 511 (1939).

# Practical Emulsions

# METAL CLEANER

Fatty Acid	8	Fatty Alcohol Sulfate	
Alcohol	15	("Wetanol")	2
Ammonia (sp. g. 0.91)	4	Neuburg Chalk 6	50
White Spirit	58		

#### SOAP PASTE PAINT CLEANER

#### FORMULA NO. 1.

Soap Chips	20.00	Water	69.30
Mineral Spirits	10.00	Sassafras Oil 7	.07

# FORMULA No. 2.

a) Naphtha	½ gal.	b) Trisodium Phosphate	4 oz.
Diglycol Stearate	1 lb.	Water	3 qt.

Heat a) on a water bath and stir until dissolved. Heat b) to a boil and run into a) slowly while mixing vigorously.8

#### PAINTED WOODWORK CLEANER

#### FORMULA No. 1.

Diglycol Stearate	1 lb.	Trisodium Phosphate	41/2	oz.
Kerosene	¼ gal.	Water	12	nt.

The diglycol stearate and kerosene are heated together in a double boiler until the stearate is thoroughly dissolved. The trisodium phosphate is dissolved in the water and heated in another container to a temperature of about 150°F. The hot water solution is then added to the hot kerosene solution while stirring at high speed. Stirring should be continued at a good rate until the mixture is of even milky consistency. Mixing may be continued at a slow rate until the batch has cooled to around 85°F. This product is applied in the usual manner by rubbing with a rag or cloth.<sup>1</sup>

1 P. C.

Bennett, Chem. Formulary, Vol. 5, p. 574 (1941).
 Ibid., Vol. 3, p. 325 (1936).

<sup>8</sup> Ibid., Vol. 4, p. 511 (1939).

#### FORMULA No. 2.

Diglycol Stearate	1	1b.	Trisodium Phosphate	4½ gal.
Kerosene		gal.	Water	3/4 gal.

Melt the diglycol stearate in the kerosene on a water bath (double boiler). In a separate container dissolve the trisodium phosphate in the water and heat to 150°F. Add the hot water solution to the hot kerosene solution while stirring at high speed. Stirring should be continued until the batch has cooled to 85°F. Rapid mixing is essential. This cleaner quickly removes dirt from paint when applied with a cloth. Upon rubbing the cleaned surface acquires a good natural luster, due to the thin colorless protective coating of diglycol stearate. This product cleans and shines in one operation.<sup>2</sup>

#### FORMULA No. 3.

DUPONOL LS Paste or		Hexalin	4.8
DUPONOL ES Paste	47.6	Tetralin	47.6

Prepare by adding the DUPONOL LS Paste to the previously mixed solvents.<sup>3</sup>

#### FORMULA No. 4.

DUPONOL LS Paste	15	Tetralin	70
Hexalin	2	Water	10
Pine Oil	3		

Prepare by mixing together at room temperature the DUPONOL, Hexalin and Pine Oil. Stir until a clear mixture is formed. Continue stirring and add the Tetralin slowly. After the Tetralin has been added and the mixture thoroughly agitated, add the water and stir until a homogeneous milky emulsion is formed.<sup>4</sup>

<sup>&</sup>lt;sup>2</sup> P. C.

<sup>&</sup>lt;sup>3</sup> E. I. Du Pont de Nemours & Co., Wilmington, Del.

E. I. Du Pont de Nemours & Co., Wilmington, Del.

#### RUG CLEANERS

# FORMULA No. 1.

Di-Glycol Oleate	44	Oleic Acid	11
Butyl Cellosolve	5	Ammonium Hydroxide	11
Ethylene Dichloride	12	Water	45
Alcohol	15		

This may be made thinner by increasing the amount of water.<sup>1</sup>

# FORMULA No. 2.

a) Oleic Acid	35	b) Butyl Cellosolve	5
Trigamine	15	Ethylene Dichloride	13
Water	125	c) Diethylene Glycol	15
		Isopropyl Alcohol	20

Mix a) and add b) with high speed agitation. Add c) slowly.<sup>2</sup> Continue stirring,

#### FORMULA No. 3.

Oleic Acid	28	Triethanolamine	15
Butyl Cellosolve	5	Water	125
Ethylene Dichloride	13	Isopropanol	14

The oleic acid, ethylene dichloride and butyl cellosolve are mixed and then added to a solution made of the triethanolamine and water. The mixture is stirred well and sufficient isopropanol is added to form a clear solution.<sup>3</sup>

#### UPHOLSTERY CLEANSER

Coal-Tar Naphtha	27 oz.	Triethanolamine Oleate	1 oz.
Tallow Soap	1 oz.	Water 4	71 07

#### SADDLE SOAP

## FORMULA No. 1.

<b>D</b>	=		
Beeswax	500	Water	800
Caustic Potash	80		

<sup>&</sup>lt;sup>1</sup> Bennett, Chem. Formulary, Vol. 1, p. 77 (1933).

<sup>&</sup>lt;sup>2</sup> Ibid., Vol. 4, p. 496 (1939).

<sup>&</sup>lt;sup>3</sup> Bennett, Chem. Formulary, Vol. 1, p. 85 (1933).

<sup>4</sup> Ibid., Vol. 4, p. 497 (1939).

Boil for 5 minutes while stirring. In another vessel heat

Castile Soap 160 Water 800

Mix the two with good stirring; remove from heat and add

Turpentine

1200

while stirring well.5

# FORMULA No. 2.

Carnauba Wax	54	Turpentine	21
Soap Flakes	20	Sperm Oil	6
Tallow	26	Water 6	5

### WASHING COMPOUND

Paraffin Wax	8 lb.	Potassium Carbonate	4 oz.
Soap, White	4 lb.	Water	l gal.
Borax	16 oz.		

Dissolve the borax and carbonate in one-third of the water by heat. Shave the soap and dissolve it in the rest of the water, adding the borax and carbonate solution after the soap has softened. Melt the wax by heating and, while hot, pour into it gradually, the hot soap solution. Remove from the fire, continue stirring until cold.<sup>1</sup>

#### COMPOSITION TO CLEAN WOODEN SURFACES

Montan Wax, Double Bleached	4	Water	68
Paraffin Wax (50/52°C.)	6	Methyl Alcohol	20
Soan Castile	1	Ammonia (Concentrated)	1

Color with any water soluble color.2

#### CLEANER EMULSION FOR PRINTING ROLLS

a) Oleic Acid Caustic Soda		b) Alcohol c) Trichlorethylene	45.5 900.0
Water	26.5	•	

<sup>&</sup>lt;sup>5</sup> Bennett, Chem. Formulary, Vol. 1, p. 84 (1933).

<sup>&</sup>lt;sup>6</sup> Ibid.

<sup>&</sup>lt;sup>1</sup> Crowley, Money Making Formulas, p. 90 (1939). <sup>2</sup> P. C.

Saponify a) to give a clear soap gel. Dissolve by working in b); stir steadily and add c) slowly, until completely mixed. Dilute with water for use.

# CLEANSER FOR RUBBER RUGS

Oleic Acid	_	Water	<b>30</b>
Triethanolamine		Spindle Oil	40

Bennett, Chem. Formulary, Vol. 4, p. 497 (1939).
 Bennett, Chem. Formulary, Vol. 4, p. 514 (1939).

# CHAPTER VI

# COSMETIC AND DRUG EMULSIONS

.....

# ALL PURPOSE CREAMS

#### FORMULA No. 1.

1. Parachol (Absorption Base)	25	5. Ozokerite, White	6
2. Lanolin, Anhydrous	10	6. Glycol Bori-Borate	2
3. Mineral Oil	20	7. Water	50
4. Paraffin Wax	2	•	

Melt together 1, 2, 3, 4, and 5 on a water-bath and keep at 50°C. Add solution of 6 in 7 heated to 50°C. a little at a time, using a low speed mixer with large paddles. Do not add additional amounts of water until first addition has been absorbed. Stir until temperature falls to 42°C. and then mix in a little perfume. Then pass through an ointment or colloid mill.<sup>1</sup>

# FORMULA No. 2.

10 20 3 5	Beeswax Ceresin Wax Water <sup>2</sup>	4 10 48
	10 20 3 5	20 Ceresin Wax

# FORMULA No. 3.

Beeswax	6.00	Rancidex (Preservative)	0.10
Spermaceti	2.00	Water	25.00
Petrolatum	5.00	Borax	0.60
Raisin Seed Oil	50.60	Triethanolamine	0:20
Mineral Oil	10.00	Perfume	0.50

<sup>&</sup>lt;sup>1</sup> Bennett, Chem. Formulary, Vol. 4, p. 52 (1939).

<sup>2</sup> Ibid.

Melt fats and oils at 160°F. Water and borax in separate kettle at same temperature. Emulsify by adding water to oils at 160°F. with constant stirring. Add perfume oils at 115°F.3

### FORMULA No. 4.

* Cosmetic Base	22	Beeswax (bleached)	7
Mineral Oil	25	Water	43
Lanolin	3		

<sup>\*</sup> The cosmetic base may be made up of 10% sorbitol mono-oleate and 90% petrolatum or 10% sorbitol di-stearate and 90% petrolatum.

S	KIN LC	TIONS	
I	ORMULA	No. 1.	
Mineral Oil Camphor	50 2	Diglycol Laurate	20
Mix together until dis	solved	and add with good stirring	5
Water 5	120		
I	ORMULA	No. 2.	
Glyceryl Monostearate Water Glycerin	6.0 75.5 10.0	Stearic Acid Oleic Acid <sup>6</sup>	1.0 2.0
I	FORMULA	No. 3.	
Stearic Acid, Triple Pressed White Mineral Oil Triethanolamine		Glycerin Perfume Water <sup>8</sup>	4 ½ 80
F	ORMULA	No. 4.	
* Base Emulsion Distilled Water	550 445	Perfume	5
*	Base E	mulsion	
Distilled Water	710	Citric Acid	10

Mineral Oil

Dried Pectin

180

50

Extract Chamomile Flowers

50

<sup>&</sup>lt;sup>3</sup> Ibid., p. 49

<sup>4</sup> Ibid.

<sup>&</sup>lt;sup>5</sup> Ibid., Vol. 5, p. 61 (1941).

<sup>6</sup> Ibid., Vol. 2, p. 146 (1939).

<sup>8</sup> Bennett, Chem. Formulary, Vol. 2, p. 147 (1935).

Moisten the pectin with a little alcohol and then rub with a little water in which the citric acid is dissolved until a fine mucilage is obtained. The pectin swells to a large extent. In the rest of the water dissolve the liquid chamomile extract and the warm solution a little at a time to the pectin mucilage. When all the water has been added, heat until a uniform solution results, avoiding overheating. The oil is then emulsified with this solution, preferably in a colloid mill or a homogenizer.<sup>9</sup>

	FORMULA	No. 5.	
<ol> <li>Mineral Oil</li> <li>Olive Oil</li> <li>Trihydroxyethylamine</li> </ol>	<b>70</b> 8	4. Water 5. Perfume 10	70 2
Stearate (Special)	14		
	FORMULA	No. 6.	
Stearic Acid Alcohol Mineral Oil Triethanolamine Glycerin		Water Quince Seed Perfume Preservative 11	75.85 3.50 .50 .15
	FORMULA	No. 7.	
Water, Distilled Sesame Oil	74 20	Citrus Pectin Citric Acid	5 1

Vigorous stirring of an acidified 5% pectin solution with 20% Sesame oil yields a stable viscous emulsion which readily disperses when poured into water. If such emulsions are homogenized and kept in closed containers, they will remain stable for years.<sup>12</sup>

#### FORMULA NO. 8.

1. Mineral Oil	72	1ъ.	3. Water (Warm)	160	lb.
2. Trihydroxyethylamine			4. Perfume		1Ъ.
Stearate (Special)		1b.			

<sup>&</sup>lt;sup>9</sup> Ibid., Vol. 3, p. 76 (1936).

<sup>&</sup>lt;sup>10</sup> Ibid., Vol. 2, p. 136 (1935).

<sup>&</sup>lt;sup>11</sup> Ibid, p. 144.

<sup>12</sup> Bennett, Chem. Formulary, Vol. 6.

Heat (1) and (2) until just melted together, and stir. Next add (3) slowly with thorough stirring and continue until the batch is homogeneous. Allow to stand one night and stir for 15 minutes before packing.

This cream washes off easily with cold water. The consistency can be changed by varying the amount of water in this formula.<sup>13</sup>

	FORMUL	No. 9.	
1. Lanolin Almond Oil Stearic Acid Diglycol Stearate Vitamin F (50,000 units per g.)		Vegetable Lecithin 2. Triethanolamine Glycerin Water 3. Perfume 14	1 20 30 800 to suit
	FORMULA	No. 10.	
Beeswax, White Quince Seed Stearic Acid		Borax Glycerin Water <sup>15</sup> No. 11.	2.50 3.00 85.45
Glyceryl Monostearate Cocoa Butter Glycerin	10 2 5	Anhydrous Lanolin Stearic Acid Water 16	2 1 80

Add the ingredients to the water, turn on the heat and bring to the boiling point with constant stirring. When all the ingredients are melted and a clear emulsion is formed, shut off the heat.

# FORMULA No. 12.

a.	Lanolin Mineral Oil Trihydroxyethylamine Stearate (Special)	12 20 41/4	b. Glycerin Water c. Benzoate of Soda Perfume 17	8 200 - <del>1/4</del> to suit
	Glycosterin	2.		

<sup>&</sup>lt;sup>13</sup> Ibid., Vol. 1, p. 109 (1933).

<sup>14</sup> Ibid., Vol. 4, p. 59 (1939).

<sup>15</sup> Bennett, Chem. Formulary, Vol. 2, p. 144 (1935).

<sup>&</sup>lt;sup>16</sup> Ibid., p. 145. <sup>17</sup> Ibid., Vol. 1, p. 122 (1933).

Cosmetic	and D	rug Emulsions	255
	FORMULA	No. 13.	
Spermaceti White Beeswax Glycerin Soap Borax Sodium Benzoate	3.00	Quince Seed Alcohol Water Perfume Preservative 18	3.40 4.00 80.00 .50
	FORMULA	No. 14.	
Spermaceti White Beeswax Glycerin Pulverized Neutral White Soap	3.75 .75 2.00		.35 3.00 2.50
	Formula	No. 15.	
<ol> <li>Mineral Oil</li> <li>White Wax</li> <li>Trihydroxyethylamine</li> </ol>	35 2	4. Perfume (Almond) 5. Water	1 5 <b>0</b>

Heat Nos. 1, 2 and 3 together to 140°F. and stir until homogeneous. Heat No. 5 to 140°F. and run in slowly to the above mixture, stirring thoroughly. When the temperature has dropped to 105°F. add the perfume drop by drop, stirring until completely absorbed. Continue stirring until cool and package.20

1 0			
	FORMULA	No. 16.	
Peanut Oil Lanolin "Moldex" (Preservative)	100 20 1	Triethanolamine Oleate Water <sup>21</sup>	2–4 200
	FORMULA	No. 17.	
Raisin Seed Oil Stearic Acid (Triple Pressed) "Rancidex"	17.50 5.50 0.10	Water Triethanolamine Alcohol Perfume	70.00 1.00 5.50 0.40

Stearate (Special)

<sup>&</sup>lt;sup>18</sup> Ibid., Vol. 2, p. 144 (1935).
<sup>19</sup> Bennett, Chem. Formulary, Vol. 2, p. 144 (1939).

<sup>&</sup>lt;sup>20</sup> Ibid., Vol. 1, p. 123 (1933). <sup>21</sup> Ibid., Vol. 4, p. 56 (1939).

Melt oil, stearic acid and preservatives at 155°F. Bring water and triethanolamine to same temperature. Add oils to water at 155°F. with slow stirring. Stir until cold. Add alcohol and perfume last.<sup>22</sup>

# FORMULA No. 18.

Lanolin Glycosterin Dextrin	2 19 4	Glycol Bori-Borate Water <sup>23</sup>	3 72
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# FORMULA No. 19.

a. Myristyl Alcohol	30	b. Glycerin	7
Petroleum Jelly	9	Water	30
2 0010100111 ) 017	-		

Melt a on the waterbath. Heat b to  $60^{\circ}$ C., and add it to a with agitation. Stir till cooled.<sup>24</sup>

# BEAUTY MILK

Mineral Oil	12.0	Stearic Acid	5.0
Grapested Oil	6.0	Glycerin	4.0
Cetyl Alcohol	1.0	Distilled Water	70.00
Triethanolamine	2.0	Perfume 1	To suit

#### ACID SKIN MILK

Olive Oil (Deodorized)	10	Acimul	12
Paraffin Wax	6	Water	. 110
Liquid Paraffin	5	Lemon Juice	3
Petroleum Jelly	10	Preservative 2	To suit

#### BATH MILK

Triethanolamine	1	Acacia or Quince Seed	
Stearic Acid	5	Mucilage	8
Glycerin or Diethylene Glycol	6 ·	Perfume Compound	To suit
Tincture Benzoin (6%)	8	Distilled Water 3	72

<sup>&</sup>lt;sup>22</sup> Ibid, p. 55.

<sup>&</sup>lt;sup>23</sup> Bennett, Chem. Formulary, Vol. 4, p. 49 (1939).

<sup>&</sup>lt;sup>24</sup> Ibid., p. 54.

<sup>&</sup>lt;sup>1</sup> Bennett, Chem. Formulary, Vol. 5, p. 62 (1941).

<sup>&</sup>lt;sup>2</sup> Ibid.

<sup>3</sup> Ibid.

# BATH OIL

Sulphonated Oil	75	Diethylene Glycol	10
Perfume Oil	15		

Distilled water may be added if desired, although a highly concentrated bath oil is usually to be preferred.4

#### ALMOND OIL EMULSION

Almond Oil	81	Oleic Acid.	6
Triethanolamine Oleate	6	Water 5	81

# LANOLIN EMULSION

Diglycol Oleate (light) 10 Lanolin (Anhydrous) 30

Warm until dissolved. Add to the above with rapid agitation

Water 60

made slightly alkaline with caustic soda (1/4%). Stir five to ten minutes.6

# Cleansing Creams

#### FORMULA No. 1.

Lanolin White Mineral Oil White Petroleum Jelly		Distilled Water Perfume <sup>1</sup>	42 To suit
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#### FORMULA No. 2.

1. Mineral Oil (White)	54.0	4. Borax	1.0
2. Beeswax	18.0	5. Water	21.0
3. Parachol	5.5	6. Perfume	0.5

Melt together 1, 2 and 3. Dissolve 4 in 5 and heat to boiling. Add this to first mixture, slowly, with stirring; add perfume before solidification begins.<sup>2</sup>

<sup>4</sup> Ibid.

<sup>&</sup>lt;sup>5</sup> Ibid., Vol. 1, p. 153 (1933).

<sup>&</sup>lt;sup>6</sup> Bennett, Chem. Formulary, Vol. 1, p. 162 (1933).

<sup>&</sup>lt;sup>1</sup> Bennett, Chem. Formulary, Vol. 3, p. 66 (1936).

<sup>&</sup>lt;sup>2</sup> Ibid., Vol. 1, p. 106 (1933).

#### FORMULA No. 3.

1. Mineral Oil	80 4. Water	90
2. Spermaceti	30 5. Glycerin	10
3. Glycosterin	24 6. Perfume to suit.	

Heat 1, 2 and 3 to 140°F. and stir into it, slowly, 4 and 5 heated to same temperature. Add perfume, at 105°F. stir slowly until cold after allowing to stand for 5 minutes. Stir until smooth and pack.3

# FORMULA NO. 4.

1. Mineral Oil	78	2. Perfume	1
White Wax	5	3. Glycerin	4
Spermaceti	28	Water	92
Trihydroxyethylamine			
Stearate (Special)	20		

Heat No. 1 and 3 separately to 200°F.; then add No. 1 to 2 slowly, stirring thoroughly. When the cream begins to set, the perfume is added and stirred in. Allow to stand over night. Stir thoroughly the next morning and package.4

## FORMULA NO. 5.

Stearic Acid	29	Triethanolamine	3.6
Lanolin (Anhydrous)	8	Carbitol	10
Mineral Oil (White)	50	Water	100

Melt the stearic acid in the mineral oil, add the lanolin and bring the temperature of this oil solution to 70°C. Then add it to the solution of triethanolamine and water which has been brought to the boiling point in a separate container. Stir vigorously to obtain a uniform emulsion and add the Carbitol solution of the perfume. Continue with even stirring until a smooth cream is obtained and then occasionally until cold. Too rapid stirring causes an undesirable aeration of the cream.5

<sup>3</sup> Ibid.

<sup>&</sup>lt;sup>4</sup> Ibid., Vol. 2, p. 133 (1935). <sup>5</sup> Ibid., Vol. 1, p. 10.

#### FORMULA No. 5A.

Ammonium Stearate (Paste)	250 oz.	Mineral Oil, White Perfume to suit.	25 oz.

# Stir until most of the ammonia has evaporated.5a

	FORMULA No. 6	FORMULA No. 7		Formula No. 6	Formula No. 7
Stearic Acid Paraffin Mineral Oil Petrolatum	8.0 7.0 30.0	8.0 35.0 10.0	Ceresin Amino Glycol Glycerin Water <sup>6</sup>	2.0 3.0 50.0	3.0 5.0 39.0
		Formula	A No. 8.		
Sorbitol Mono Sorbitol Di-Si Beeswax		5 <b>10</b> 7	Mineral Oil Lanolin Water		32 <b>3</b> 43

This combination gives a moderately firm cream with a fine-grained texture.8

# FORMULA No. 9.

U.S.P. Flaked White Beeswax	10	Water	40
Ceresin Wax (Melting Point		Borax	1/2
138°F.)	5	Perfume	Sufficient
Mineral Oil (Viscosity 68/75)	65		

The beeswax, ceresin and a portion of the mineral oil are placed in a steam jacketed kettle and the temperature in melting the waxes should be at no time in excess of 80°C. The emulsifying power of beeswax is retarded or broken down when the temperature is too high.

#### FORMULA No. 10.

Sorbitan Monostearate	12	Water	48
Olive Oil, Deodorised	40	Preservative 10	q.s.

<sup>&</sup>lt;sup>5a</sup> Bennett, Chem. Formulary, Vol. 1, p. 106 (1933).

<sup>&</sup>lt;sup>6</sup> Kalish, Drug & Cosmetic Industry, 48, 36 (1941).

<sup>8</sup> Bennett, Chem. Formulary, Vol. 4, p. 49 (1939).

<sup>9</sup> Ibid., Vol. 11, p. 146 (1935).

<sup>10</sup> Atlas Powder Co., Wilmington, Del.

# Practical Emulsions

	Formula	No. 11.	
Lanette Wax SX Lanette Wax K Sorbitan Mono-Oleate Raisin-Seed Oil	12 6 2 10	Sorbitol Syrup Water Preservative <sup>11</sup>	5 65 q.s.
	Formula	No. 12.	
Glyceryl Monostearate Liquid Paraffin Stearyl Alcohol Sorbitan Mono-Oleate	14 10 1 1	Sorbitol Syrup Water Preservative 12	5 69 q.s.
	Formula	No. 13.	
Liquid Paraffin Stearic Acid Isopropanolamine Sorbitan Monostearate	10.0 3.3 1.7 2.0	Sorbitan Mono-Oleate Water Preservative <sup>18</sup>	2.0 79.0 q.s.
	Formula	No. 14.	
Sorbitol Mono-Oleate Sorbitol Monostearate Sorbitol Syrup Stearyl Alcohol		Isopropanolamine Water	1.0 0.5 94.5 q.s.
	Formula	No. 15.	
Mannitan Monolaurate Olive Oil, Deodorized	10.0 30.0	Water Preservative <sup>15</sup>	60.0 q.s.
	Cold C	reams	

# FORMULA No. 1.

1. Diglycol Stearate	14 1ь.	4. Petrolatum (White)	6 1b.
<ol><li>Paraffin Wax</li></ol>	2 lb.	5. Water	6 gal.
3. Mineral Oil	33/4 gal.	6. Perfume Oil	5½ fl. oz.

a Melt Nos. 1, 2, 3 and 4 at 170°F.

b Heat 5 to 180°F.

<sup>11</sup> Ibid.

<sup>12</sup> Ibid.

<sup>18</sup> Ibid.

<sup>14</sup> Ibid.

<sup>15</sup> Ibid.

- c Add b to a while mixing. Allow mixer to run until batch is completely emulsified.
- d Allow batch to cool to 125°F. and add 6 and mix at low speed.
- e Batch should be allowed to cool without stirring to 105°F. at which temperature it is poured into jars.

## FORMULA No. 2.

Stearic Acid Butyl Stearate Amino Glycol	15 5 2	Zinc Stearate Water <sup>2</sup>	15 63
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# FORMULA No. 3.

Glyceryl Monostearate	8	Petrolatum	10
Magnesium Stearate	14	Mineral Oil, White	5
Beeswax	3	Water <sup>3</sup>	60

#### FORMULA No. 4.

A very low priced light bodied greaseless but stable cream is made as follows:

Glycosterin	22		∫ Water		128
Petrolatum, White	16	2.	Borax		3
Paraffin Wax	12		Potassium	Carbonate	2
Mineral Oil	32		•		

Heat above separately to 80°C. and pour (2) into (1) slowly while stirring. Add perfume at 55°C. stir and pack. If cold packed a high gloss is given to surface by passing a flame lightly over surface in each jar.<sup>4</sup>

#### FORMULA No. 5.

Cetyl Alcohol	100	Cholesterin	5
White Beeswax	150	Borax	10
Vegetable Oil	200	Water	260
Mineral Oil	250	Perfume 5	25

<sup>&</sup>lt;sup>1</sup> Bennett, Chem. Formulary, Vol. 3, p. 65 (1936). <sup>2</sup> Kalish, Drug & Cosmetic Industry, 48, 36 (1941).

<sup>&</sup>lt;sup>8</sup> Ibid., Vol. 3, p. 67 (1936).

<sup>4</sup> Ibid., Vol. 1, p. 108 (1933).

<sup>&</sup>lt;sup>5</sup> Goldschmidt, U. S. 2,269,529 (1942).

#### FORMULA No. 6.

Stearic Acid Esters of		Mineral Oil	15
Polyethylene Glycol	10	Petrolatum	10
Stearic Acid	10	Salt, such as Sodium Chloride	6
Paraffin Wax	15	Water,5 To make	100

## FORMULA No. 7.

## COSMETIC CREAMS

Stearyl Alcohol	1.0	Petroleum Jelly	45.0
Mannide Monooleate	3.5	Water 6	45.5
Raisin Seed Oil	5.0		

# FORMULA No. 8.

Beeswax	540	Water	720
Spermaceti	300	Borax	100
Mineral Oil	1730	Sodium Benzoate	10
Stearin	430	Perfume.	

The fat bases should be melted with mineral oil. The borax and benzoate of soda is dissolved in water and brought to the boil and stirred while still hot into the molten fats. Allow to cool with slow agitation. Add perfume.

#### FORMULA No. 9.

a. Ceresin, White	27	Mineral Oil, White	87
Spermaceti	25	b. Borax	5
Stearic Acid	21	Water, Distilled	36
Undecvlic Acid	1	-	

Melt a and add with agitation to the hot solution b. Stir until cold.

#### FORMULA No. 10.

Cetyl Alcohol	10	Vaseline, White	
Paraffin, Liquid	10	Water	60

Transparent, soft, white cream.9

<sup>5</sup> P. C.

<sup>&</sup>lt;sup>6</sup> Kalish, Drug and Cosmetic Industry, 48, 36 (1941).

<sup>&</sup>lt;sup>7</sup> Bennett, Chem. Formulary, Vol. 1, p. 109 (1933).

<sup>&</sup>lt;sup>8</sup> Ibid., Vol. 4, p. 48 (1939).

<sup>&</sup>lt;sup>9</sup> Bennett, Chem. Formulary, Vol. 3, p. 65 (1936).

#### FORMULA NO. 11.

Mineral Oil	1 gal.	Water (preferably	
White Beeswax	2 lb.	distilled)	1/2 gal.
		Powdered Borax (bolted)	2 oz.

Mix beeswax and oil in one container. Bring to 150°F. then reduce to 120°F. Dissolve borax in water. Bring to 120°F. Pour borax and water solution slowly into wax and oil solution stirring constantly but not rapidly. At 115°F., perfume and pour into containers.10

Formu	la No.	12.				
Stearic Acid 16 Glycerin 48 Mineral Oil 12 Paraffin Wax	Wa	onger Ammor ter fume <sup>11</sup>	ia Water	4 64		
Formula No	. 13	14	15	16		
Sorbitol Syrup	3.0. 0.7	8.0 1.0	5.0 0.7	2.5		
Triethanolamine		1.0		1.7		
Water	76.4	64.1	45.1	70.9		
Preservative ("Moldex")	0.1	0.1	0.1	0.1		
Stearic Acid	15.0	20.0	10.0	3.5		
Lanolin	0.5	1.5	0.5	2.0		
Mineral Oil	2.0	2.0	35.3	15.0		
Mannitan Monoöleate	2.0	1.0	1.0	2.0		
Mannitan Monostearate			2.0	2.0		
Spermaceti		2.0				
Perfume 12	0.4	0.4	0.4	0.4		
Formula No. 17.						
Spermaceti 125	Bor	ax		5		
White Wax 120		tilled Water		190		
Liquid Petrolatum 560		of Rose, Syn	thetic 13	q.s.		

<sup>10</sup> Ibid., Vol. 1, p. 109 (1933).11 Ibid., Vol. 3, p. 65 (1939).

<sup>12</sup> Atlas Powder Co., Wilmington, Del.

<sup>13</sup> Bennett, Chem. Formulary, Vol. 1, p. 108 (1933).

#### FORMULA No. 18.

Stearic Acid of Polyethyler Glycol Spermaceti Glycerine	ne 15 5 3	Aluminum Chlorid Crystals Water <sup>14</sup>		ated 12 make 100
	FORMULA	No. 19.		
Zinc Sulphate Alum Acacia (Powdered) White Beeswax Glycerin	0.15 0.2 4.0 15.5 13.0	Sodium Benzoate Cocoa Butter Perfume Distilled Water <sup>15</sup>		0.1 3.0 0.5 63.53
Formula No. 20	21	FORMULA	No. 20	21
Stearic Acid 8 Paraffin 7 Mineral Oil 30 Petrolatum	8 35 10	Ceresin Amino Glycol Glycerin Water <sup>16</sup>	2 3 50	3 5 39
	FORMULA	No. 22.		
Stearic Acid Cetyl Alcohol Butyl Stearate	4.0 1.0 3.0			0.8 0.5 90.7
	FORMULA	No. 23.		
Stearic Acid Lanolin (Anhydrous) Beeswax (White) Mineral Oil (White)	30 20 16 33	Triethanolamine Carbitol Water <sup>18</sup>		3.8 16 95
	FORMULA	No. 24.		
White Mineral Oil White Beeswax U.S.P.	73.5 16.5	Paraffin Wax Ozokerite		5.0 5.0

The above should be heated to a temperature slightly above its melting point. The proper type of perfume is then added.

In a separate container, 1½ pound borax is dissolved in water. The amount of water can vary from 26 to 36 pounds per 100 pounds of finished cold cream.

<sup>14</sup> Goldschmidt, U. S. 2,269,529 (1942).

<sup>Bennett, Chem. Formulary, Vol. 4, p. 49 (1939).
Kalish, Drug and Cosmetic Industry, 48, 36 (1941).</sup> 

<sup>18</sup> Bennett, Chem. Formulary, Vol. 1, p. 107 (1933).

The borax dissolved in the water and heated almost to the boiling point (approximately 200°F.) is then added all at one time to the above mixture. Stir during the addition of the borax water and continue stirring until a complete emulsion has been formed. The cream should be filled into the jars while still warm; not hot but just warm enough to pour easily so that it will have a perfectly smooth and even appearance at normal temperature.

Caution: If the water is not hot enough when added to the base, the cream will separate drops of water upon standing. If the stirring is not properly done or not continued long enough, the cream will likewise separate water.<sup>19</sup>

# FORMULA No. 25.

White Mineral Oil	45.0	Spermaceti	6.0
White Beeswax U.S.P.	13.0		

Heat all together until liquid; then add under stirring

Powdered Borax 1.0 Water 20 3	35.0
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which has been heated previously to about 85°C.

#### FORMULA No. 26.

Apricot Kernel Oil	54	Ethyl Amino Benzoate	1/2
White Beeswax	13	Borax Powder	1/2
White Ceresin Wax	81/2	Distilled Water	25

Melt apricot kernel oil, beeswax and ceresin wax together and add ethyl amino benzoate. Stir until dissolved. Adjust temperature to 65°C. Dissolve borax in hot distilled water and filter. Adjust temperature to 65°C. Then add borax solution slowly while stirring to the oil and wax mixture kept at the same temperature and stir until cold.<sup>21</sup>

<sup>&</sup>lt;sup>19</sup> Ibid., Vol. 2, p. 147 (1935).

<sup>20</sup> Ibid.

<sup>&</sup>lt;sup>21</sup> Ibid., Vol. 1, p. 109 (1933).

# FORMULA No. 27.

Beeswax	15.0	Water	20.0
Petrolatum	14.0	Borax	0.5
Mineral Oil	50.0	Perfume	0.5

Melt wax and petrolatum. Add oil. Dissolve borax in hot water. Add to above with stirring.22 Perfume at 120°F.

#### FORMULA No. 28.

a. Beeswax, White	60	c. Borax	5
Spermaceti	70	Benzoic Acid <sup>23</sup>	5
Stearic Acid	10	Water	220
b. Peanut Oil	620	d. Perfume	10

Melt a, and mix with b. Take to  $55^{\circ}$ C. on the waterbath, and add the hot solution c in small portions with good agitation. When properly emulsified and slightly cooled, add c. Stir until cold.

#### FORMULA No. 29.

Glycosterin	20	Mineral Oil	8
Paraffin Wax	30	Water	200
Petrolatum White	18	Perfume 24	To Suit

#### FORMULA No. 30.

Spermaceti	125	Borax	5
White Wax	120	Water, Distilled 25	190
Liquid Petrolatum	560	•	

#### FORMULA No. 31.

Stearic Acid Paraffin Wax	4	Glycerine	12
I ai aiiiii Wax		Add Ammonia (26°)	½ oz.

When there is a perfect saponification, add 16 oz. warm distilled water in which must be dissolved 15 grams powdered horax.26

<sup>&</sup>lt;sup>22</sup> Bennett, Chem. Formulary, Vol. 2, p. 147 (1935).

<sup>&</sup>lt;sup>23</sup> Ibid., Vol. 4, p. 48 (1939).

<sup>&</sup>lt;sup>24</sup> Ibid., Vol. 1, p. 108 (1933). <sup>25</sup> Ibid., p. 109.

<sup>26</sup> Ibid.

#### FORMULA No. 32.

White Wax	10 oz.	Liquid Petrolatum	3 1b.
Paraffin	9 oz.	Borax	1 oz.
Ceresin	2 oz.	Water, Distilled 27	pt., 4 fl. oz.
White Petrolatum	8 oz.		

## FORMULA No. 33.

a. Beeswax, White	80	Benzoic Acid	5
Spermaceti	50	c. Perfume	9
Stearic Acid	20	d. "Apple Ether"	1
Lanolin, Anhydrous	20	e. Peanut Oil	540
b. Borax	5	f. Water	270

Melt a, mix in e, and saponify with b and f. Add d to cover the odor of the lanolin; perfume with c.<sup>28</sup>

#### FORMULA No. 34.

Hard Paraffin Wax	50	Lanolin, Anhydrous	60
Petrolatum, Yellow	400	Rosin, Pale	40
Peanut Oil	200	Water	140
Beeswax, Yellow	100	Borax 29	10

#### FORMULA No. 35.

Parachol (Absorption Base)	125	Glycerin	200
Peanut Oil	400	Water 30	800

#### FORMULA No. 36.

a. Woolfat, Anhydrous,		Peanut, Olive, or Sesame O	il 20
Neutral	40	b. Glycerin	5
Petrolatum, White	10	Water	25

Melt a on waterbath. Add b. Stir till cold. 31

# Perspiration Deodorizing Creams

#### FORMULA No. 1.

Beeswax	8 oz.	Benzoic Acid	20 gr.
Liquid Petrolatum	24 oz.	Salicylic Acid	400 gr.
Sodium Borate	100 gr.	Hot Water	16 oz.

<sup>&</sup>lt;sup>27</sup> Ibid., Vol. 1, p. 108 (1933).

<sup>&</sup>lt;sup>28</sup> Bennett, Chem. Formulary, Vol. 4, p. 48 (1939).

<sup>&</sup>lt;sup>29</sup> Ibid., p. 49.

<sup>80</sup> Ibid., p. 56.

<sup>81</sup> Ibid., Vol. 4, p. 51 (1939).

Melt the wax and oil and heat to about 160 degrees F. Dissolve the other materials in the water, heat to the same temperatures as the wax solution, and pour it into the latter, beating briskly until the cream is formed. Here a comparatively high temperature of the solutions, plus a small amount of stirring, results in a glossy cream.<sup>1</sup>

## FORMULA No. 2.

Mannide Mono-oleate	2	Water 4	0
Petrolatum	38	Hydrated Aluminium Chloride 2 2	0:

#### FORMULA No. 3.

Paraffin Wax	10	Petrolatum	5
Mineral Oil	10	Aluminum Sulphate	10
Deomul	15	Water	50

Heat waxes and oils to 70°C., add 30 parts water. Cool to 45°C., and stir in salt dissolved in 20 parts water.<sup>3</sup>

#### FORMULA No. 4.

Polymerized Glycol Stearate	15	Aluminum	Chloride,	Hydrated	
Spermaceti	5	Crystals	•		12
Glycerin	3	Water 4		To make	100

#### FORMULA No. 5.

a. Mineral Oil	25	c. Oxyquinoline Sulphate	
Acimul -	25	(0.1% Solution)	25
b. Water	25		

a is heated till clear. b is heated to 75°C. and added to a stirring thoroughly. At 60°C. add c and stir. It is necessary to add c in small portions taking care to make no more additions until the previous portions have been emulsified. Add perfume. Stir. Pour at 55°C.

<sup>2</sup> Atlas Powder Co., Wilmington, Del.

<sup>&</sup>lt;sup>1</sup> Ibid., Vol. 1, p. 115 (1933).

<sup>&</sup>lt;sup>8</sup> Bennett, Chem. Formulary, Vol. 5, p. 58 (1941).

<sup>&</sup>lt;sup>4</sup> Glyco Products Co., Inc., Brooklyn, N. Y.

<sup>&</sup>lt;sup>5</sup> Bennett, Chem. Formulary, Vol. 4, p. 53 (1939).

# Cosmetic and Drug Emulsions

#### DEPILATORY CREAM

a. Tragacanth, Gum	3.0	b. Isocholesterol	14.0
Glycerin	8.0	White Beeswax	4.0
Water	45.9	Mineral Oil (0.881)	25.0
Sodium Benzoate	0.1		

Disperse a in the usual manner. Cool to a medium warmth, add the mixture b in small portions. Homogenize.

# FRECKLE "PREVENTATIVE"

Quinine Bisulphate	20	Mineral Oil	100
Water	477	Woolfat	100
Amino Stearin Petrolatum, White	100 200	Perfume Oil	3

Melt the oils and waxes with agitation and add, at about 70°C., the quinine bisulphate solution of equal temperature. Stir until cold.<sup>1</sup>

#### ACID CREAMS

#### FORMULA No. 1.

Glycol Distearate, Pure	14.0	Citrus Pectin	0.5
Mineral Oil	3.0	Perfume	0.5
White Beeswax	2.0	Citric Acid	2.0
Ceresin	1.0	Glycerin	6.0
Lanolin	1.0	Distilled Water 2	69.0
Sodium Lauryl Sulphate	1.0		•

#### FORMULA No. 2.

Wool Wax	3.0	Glycerin	4.0
Lanolin	8.0	Lactic Acid	1.5
Stearyl Alcohol	2.0	Distilled Water 3	44.5
Petrolatum	37.0		

#### HONEY AND ALMOND TYPE CREAM

Cold Cream Almond Oil	• .	Solution of Soda, B.P. Quince Mucilage	1½ 5	oz. oz.
Honey	½ oz.	Water to make	5	pt.
Boric Acid	1 oz.			

<sup>&</sup>lt;sup>6</sup> Ibid., p. 61.

<sup>&</sup>lt;sup>1</sup> Ibid., p. 56.

<sup>&</sup>lt;sup>2</sup> Bennett, Chem. Formulary, Vol. 5, p. 59 (1941).

<sup>8</sup> Ibid.

Stir the cold cream, almond oil, and solution of soda together until a uniform soapy emulsion is obtained. Dissolve the boric acid in 60 ounces of warm water; to this add the glycerin and quince mucilage, and add the mixture slowly, and with constant stirring to the mortar contents. Perfume with spirits of almonds and rose when cold and make up.'

#### ALUM CREAM

Petrolatum, White	9.0	Carbitol	3.0
Paraffin Wax (52°C.)	6.5	Alum Solution, Saturated	5.5
Mineral Oil	14.0	Rose Water	50.0
Acimul	12.0		

Melt the oil and waxes at 70°C. Add them to water, alum solution and Carbitol of the same temperature with good stirring. Stir until cold enough to be just about pourable (50°C.). Perfume at 55°C.<sup>7</sup>

#### ASTRINGENT CREAMS

#### FORMULA NO. 1.

Diglycol Stearate	10	Heat to 170°F.	
Lanolin Anhydrous	5	Tannic Acid	2
Mineral Oil	3	Water	70

Heat to 170°F. and add to above at that temperature with agitation.

Perfume as desired.8

#### FORMULA No. 2.

Paraffin	15	Mineral Oil	60
Lanolin	10	Cetyl Alcohol	15

Heat to 150°. Then add a heated solution of:

<sup>4</sup> Ibid., Vol. 2, p. 146 (1935).

<sup>&</sup>lt;sup>7</sup> Bennett, Chem. Formulary, Vol. 4, p. 54 (1939).

<sup>8</sup> Ibid., Vol. 2, p. 148 (1935).

Alum	15	Water	280
Tannic Acid	15		

Agitate until the cream congeals.9

#### AVOCADO TISSUE CREAM

Avocado Oil	20	Almond Oil	25
Lanolin Absorption Base	10	Preservative	0.2
Stearic Acid	5	Perfume	0.8
Cetyl Alcohol	5	Borax	1
Beeswax	5	Distilled Water 10	28

The water and borax are brought to a temperature of 165°F, and are then added to the molten fats, oils and waxes. Careful heating and constant stirring are essential, the perfume being added when the temperature has dropped to about 120°F.

## AVOCADO OIL CLEANSING CREAM

Part 1.		Part 2.	
Hydrogenated Oil (Cosmetic	:	Distilled Water	15.3
Grade)	11.0	Borax U.S.P.	0.5
Beeswax U.S.P., White	5.0	Part 3.	
Stearic Acid, Triple Pressed	0.5	Perfume 11	0.6
Sesame Seed Oil	60.0	Procedure: Melt Part	1 at 80°C.
Avocado Oil	7.0	Heat Part	2 to 75°C.
Antioxidant & Preservative	0.1		

When Part 1 has cooled to 75°C. add Part 2 with constant stirring. Add Part 3 at 60°C. Pour between 55°-50°C.11

# ALMOND CREAM, LIQUID

Sweet Almond Oil	1 1b.	Borax	2 lb.
Spermaceti	2 1b.	Quince Jelly	1 lb.
Beeswax	2 lb.	Alcohol	1 pt.
Castile Soap, Powdered	3 1b.	Water	4 pt.

Melt the spermaceti and wax together. Dissolve the soap and borax in hot water. Mix these together and add balance of ingredients. Stir and filter through cloth.12

<sup>9</sup> Ibid.

<sup>&</sup>lt;sup>10</sup> Ibid., Vol. 4, p. 50 (1939).

<sup>&</sup>lt;sup>11</sup> Bennett, Chem. Formulary, Vol. 4, p. 49 (1939). <sup>12</sup> Ibid., Vol. 1, p. 105 (1933).

# BORO-GLYCERIN LANOLIN CREAM

a. Boric Acid Glycerin	10 g. 40 g.	<ol> <li>Lanolin, Anhydrous Vaseline, White Melt gently.</li> </ol>	100 600	0
Water Dissolve.	250 g.	c. Rose Oil, Artificial or Eau de Cologne Oil 18		cc.

## PEROXIDE CREAM

a. Paraffin Wax Mineral Oil Lanolin Acimul	10	b. Hydrogen Peroxide (100 volume) c. Water	5 60
Acimui	10		

Heat a to 70°C. and stir till completely clear. Heat c to 70°C. and add a to c slowly, stirring in one direction only. At 55°C. add b and perfume. Stir and pour at 50°C.

If 5 parts of the water are replaced by 5 parts "Carbitol" the cream can be poured at 45°C.14

# BLEACHING CREAM

a. Petrolatum (White) Paraffin Wax Mineral Oil	9 6.5 14 12	b. Carbitol * Lemon Juice Water	3 5.5 50
Acimul	12		

<sup>\*</sup>Lemon juice can be replaced by citric and lactic acid, etc., in proportionate amounts.35

A is melted, b is mixed, heated to 70-75°C. and poured slowly into a, with stirring. Perfume is added at 55°C. Pour at 50°C.

#### CHOLESTEROL-LECITHIN CREAM

#### FORMULA No. 1.

Lanolin, Anhydrous	20	Cholesterol	6
Stearin	10	Lecithin	12
Cacao Butter	20	Water	80
White Wax	20	Sodium Benzoate	1.5
Sweet Almond Oil, Preserve	ed	Borax	15
with Nipagin	200	Nipagin M 16 (Preservative)	0.8

<sup>13</sup> Ibid., Vol. 2, p. 70 (1935).

<sup>14</sup> Bennett, Chem. Formulary, Vol. 4, p. 52 (1939).

<sup>&</sup>lt;sup>18</sup> Ibid., Vol. 1, p. 104 (1933).

#### FORMULA No. 2.

Lanolin, Anhydrous	30	Water	18
White Wax	50	Cholesterol	1.5
Spermaceti	10	Egg Lecithin 17	0.5
Borax	2		

## FORMULA No. 3.

1. Parachol	10	Mineral Oil	10
Parasterin	20	2. Water	25

Heat (1) in water, both, till melted, allow to cool to 45–47°C. Warm (2) to 45–47°C. and add in 7 or 8 different portions to (1), stirring vigorously, taking care not to add more water until previous portions are absorbed.<sup>18</sup>

# FORMULA No. 4.

Lanolin (Anhydrous)	12	Cream Petrolatum (Short	
Water (Distilled)	12	Fiber)	55.5
Lecithin	0.5	Mineral Oil (White)	20
		Perfume 19	To suit

#### HORMONE CREAM

a. Petrolatum	15	Spermaceti	
Lanolin, Anhydrous	15	b. Hormone Oil	20
Absorption Base	10	c. Water or Rose Water	40
Olive Oil	5	d. Perfume	

Melt a on the waterbath. Cool to below 35°C., and add b—then emulsify with c. Perfume last.<sup>20</sup>

# LEMON JUICE CREAM

Five parts of oxy-cholesterin and 95 parts of petrolatum are thoroughly mixed to form an absorption base. Twenty parts of petrolatum and three parts of beeswax are melted together, and 30 parts of the base are added with thorough stirring. Fifty parts of natural lemon juice are added to the

<sup>17</sup> Ibid.

<sup>&</sup>lt;sup>18</sup> Ibid., p. 105.

<sup>19</sup> Bennett, Chem. Formulary, Vol. 3, p. 68 (1936).

<sup>&</sup>lt;sup>20</sup> Ibid., Vol. 4, p. 53 (1939).

above mixture while still hot and stirring is continued until the mass is cool.<sup>21</sup>

#### LIME AND GLYCERIN CREAM

Almond or Peach Kernel Oil	35.00	Lemon Oil	1.25
Glycerin	1.50	Lime Water, To make 22	80.00

# LIQUID LIP MAKE-UP

a. Triethanolamine Oleate	15	Mineral Oil, White	40
Glycerin	10	Scarlet, Oil-Soluble	2.5
Water	10	c. Perfume (Eau de Cologne	
b. Beeswax	10	Oil, Rose, Fougère)	

Emulsify by adding b to a at 50–60°C. Stir till cold, and add perfume.<sup>23</sup>

#### MASCARA

Glyceryl Monostearate	10	Beeswax	25
Triethanolamine	5	Gelatin	2
Stearic Acid	15	Water	13
White Petrolatum	20	Carbon Black	10

Make a solution of the gelatin in water, add the triethanolamine. Melt the wax, glyceryl monostearate, petrolatum and stearic acid, add the pigment and then the hot gelatin solution. Mill to get a uniform product.<sup>24</sup>

#### MOUSTACHE POMADE

Glycosterin	4	Beeswax	30
Gum Arabic	8	Diethylene Glycol	12
Water	31	Perfume to suit.	
Castile Soap	12		

Warm until wax has melted and mix vigorously until uniform.  $^{25}$ 

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Loid., Vol. 3, p. 70 (1936).
Hoid., Vol. 5, p. 59 (1941).
Loid., Vol. 4, p. 54 (1939).
Bennett. Chem. Formulary, Vol. 5, p. 63 (1941).
Hoid., Vol. 4, p. 66 (1939).
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12

# "NOURISHING" CREAMS

#### FORMULA No. 1.

Absorption Base	25	Preservative	1.3
Diglycol Stearate	15	Water	75
Olive Oil	15		

Melt absorption base, diglycol stearate, and olive oil, and add to water which has been heated to same temperature and contains preservative. Mix well and stir slowly till cool. Incorporate perfume and package.<sup>1</sup>

# FORMULA No. 2.

Mineral Oil, White	35 cc.	Amino Stearin	8 g.
Beeswax	20 g.	Water	50 cc.

Warm together and mix vigorously until emulsified.2

# FORMULA No. 3.

12 Mineral Oil

Petrolatum Lanolin	4 Water 3	65
	FORMULA No. 4.	
White Wax	600 Cacao Butter	400

000	Cacao Dunci	-100
100	Sweet Almond Oil	
500	(With preservative)	1800
600	Cholesterol, Purest	120
	100 500	100 Sweet Almond Oil 500 (With preservative)

After solution of the cholesterol has been effected, stir the following hot solution into the molten mass until pasty:

Sodium Benzoate Borax	15 100	Water <sup>4</sup>	1700
	FORMULA	No. 5.	
Beeswax	7	Water	50
Spermaceti	8	Mineral Oil	15
Lanolin Absorption Base	20		

<sup>&</sup>lt;sup>1</sup> Ibid., Vol. 2, p. 146 (1935).

Glycosterin

<sup>&</sup>lt;sup>2</sup> Glyco Products Co., Inc., Brooklyn, N. Y.

<sup>&</sup>lt;sup>3</sup> Ibid., Vol. 1, p. 112 (1933).

<sup>&</sup>lt;sup>4</sup> Bennett, Chem. Formulary, Vol. 1, p. 112 (1933).

Melt the waxes and add the mineral oil. Warm the absorption base to 40°C. and the water likewise; then slowly add the water with steady but not violent agitation. Then add the melted waxes which should be of the same temperature.<sup>5</sup>

# FORMULA No. 6.

Lanolin absorption base 400, glycerin 50, arnica (1:10) 50, witch hazel extract 150, distilled water 347, perfume 3. Heat the absorption base until soft (35°C.) and incorporate water previously heated to the same temperature a little at a time. Finally add the other ingredients. All parts by weight.<sup>6</sup>

#### FORMULA No. 7.

Cetyl Alcohol Lanolin Cocoa Butter	50	Arachis Oil Mineral Oil Petrolatum, White	100 150 240 280
Cholesterin	10	Water	280
White Beeswax	50	Perfume 7	20

#### FORMULA No. 8.

Soft Paraffin Containing		Triethanolamine	9
1% Oxycholesterol	746	Rose Water	225
Stearic Acid	20	Perfume	To suit

This cream can be made by dissolving the stearic acid and oxycholesterol in the molten soft paraffin, bringing the temperature to about 70–75°C., adding the mixture of rose water all at once at the same temperature, stirring vigorously (the heat being maintained) until the mixture is quite homogeneous, transferring to a cold mortar, and whisking until cold.<sup>8</sup>

## FORMULA No. 9.

Lanolin		Triethanolamine	5
Stearic Acid	15	Water	200

<sup>5</sup> Ibid., Vol. 2, p. 147 (1935).

<sup>6</sup> Ibid., Vol. 4, p. 52 (1939).

<sup>7</sup> Ibid.

<sup>8</sup> Ibid., p. 53.

Weigh out the triethanolamine and stearic acid and add to the whole quantity of water. Heat the mixture in a kettle and, when the stearic acid is melted, stir to a creamy soap solution. Add the lanolin and continue heating without stirring until the lanolin is melted and the mixture is just below the boiling point.

At this point stir the mixture thoroughly until a thick creamy emulsion results. Continue stirring intermittently until the emulsion has cooled to room temperature.<sup>9</sup>

#### FORMULA No. 10.

1. Diglycol Stearate	14 lb.	7. Perfume Oil 5½ fl. oz
2. Mineral Oil	33/4 gal.	8. Solution Yellow Color
3. Lanolin	6 lb.	Made by Dissolving
4. Petrolatum (White)	2 lb.	Yellow Dye 2 drams in
5. Water	6 gal.	Mineral Oil 14 fl. oz. 81/4 fl. oz
6 Turtle Oil	51/2 ft 07	•

# Method of manufacture:

- a. Melt 1, 2, 3, 4, 6 and 8 at 170°F.
- b. Heat 5 to 180°F.
- c. Add b to a while mixing. Allow mixer to run until batch is completely emulsified.
- d. Allow batch to cool to 125°F. and add 7, and mix at low speed.
- c. Batch should be allowed to cool without stirring to 100° F. at which temperature it is poured.<sup>10</sup>

# FORMULA No. 11.

White Wax	5 oz.	Water	10 oz.
Spermaceti	1 lb.	Benzyl Alcohol	1
Petrolatum (Light Amber)	1 1b.	Bitter Almond Oil	1
Mineral Oil	1½ pt.	Rose Geranium Oil	11/2
Lanolin (Hydrous)	2 lb.	Bergamot Oil 11	2
Borax	3/8 OZ.	9	_

<sup>&</sup>lt;sup>9</sup> Bennett, Chem. Formulary, Vol. 1, p. 104.

<sup>&</sup>lt;sup>10</sup> Ibid., Vol. 3, p. 70.

<sup>11</sup> Ibid., Vol. 1, p. 112.

#### FORMULA No. 12.

<ol> <li>Spermaceti</li> </ol>	10 lb.	Almond Oil	30 lb.
Lanolin	20 lb.	2. Water	90 lb.
Glycosterin	46 lb.	Sodium Benzoate	¼ lb.
Olive Oil	20 lb.	3. Perfume	to suit

Heat (1) to 150°F, and run into it slowly with stirring (2) which has been heated to the same temperature. Add the perfume at about 105°F, and stir in. Pour at 95–100°F.<sup>12</sup>

### FORMULA No. 13.

Absorption Base	30	Water	55
Lanolin	5	Beeswax, White	10

Procedure: Melt the wax and lanolin, add the base and stir in the water (warm).

(Note: Consistency in the foregoing formulas can be adjusted by changing the wax content to suit.) 13

#### FORMULA No. 14.

Lanolin, Anhydrous	Beeswax, White	40
Spermaceti	Cocoa Butter, Odorless 14	28
•	•	

#### FORMULA No. 15.

Almond Oil (With		Sodium Benzoate	5
preservative)	390	Parahydroxybenzoic Acid	2
Lecithin	50	Water 15	220
Borax	5		

#### FORMULA No. 16.

Trihydroxyethylamine Stearate	20	Carbito1	20
Turtle Oil	50	Water (Distilled)	300
Paraffin Oil (White)	40	Preservative	5
Diglycol Stearate	5	Perfume Oil	To Suit

Melt together the first four of these ingredients, and slowly add the water, which has been heated to 50°C., with

<sup>12</sup> Ibid.

<sup>13</sup> Bennett, Chem. Formulary, Vol. 1, p. 113 (1933).

<sup>14</sup> Ibid., p. 112.

<sup>15</sup> Ibid., p. 113.

constant stirring. Continue stirring until cool, and then add Carbitol, containing the perfume and preservative. 16

## FORMULA No. 17.

Part 1.		Part 2.	
Hydrogenated Oil	25.6	Water (Distilled)	15.8
Beeswax U.S.P.	9.7	Borax U.S.P.	0.5
Lanolin Anhydrous U.S.P.	8.5	Part 3.	
Spermaceti Ü.S.P.	2.5	Oleic Acid (White)	0.3
Sesame Seed Oil U.S.P.	36.5	Perfume	0.5
Preservative and Antioxidan	nt 0.1		

Melt Part 1 at 70°C. Bring Part 2 to same temperature and add to Part 1 with slow agitation. Add Part 3 at above 60°C. Pour hot and top.17

# FORMULA No. 18.

Ti-Tree Oil	10 g.	Glycerin	1 g.
Lanette Wax SX	10 g.	Water 18	50 g.
Mineral Oil	29 g.		

#### PROTECTIVE HAND CREAMS

#### FORMULA NO. 1.

Glyceryl Monostearate	8	Petrolatum	10
Magnesium Stearate	14	Mineral Oil	5
Beeswax	3	Water	60

Heat together to 70°C. and stir until cool.19

#### FORMULA No. 2.

Lanolin (Anhydrous)	20	Glycol Stearate	12
Paraffin Wax	6	Water	55
Paraffin Oil	7		

Melt lanolin, glycol stearate, and wax; add oil; then add water while agitating rapidly until cold.20

<sup>16</sup> P. C.

<sup>&</sup>lt;sup>17</sup> Ibid., Vol. 4, p. 50 (1939).
<sup>18</sup> Ibid., Vol. 5, p. 59 (1941).

<sup>&</sup>lt;sup>19</sup> Bennett, Chem. Formulary, Vol. 2, p. 146 (1935).

<sup>&</sup>lt;sup>20</sup> Glyco Products Co., Inc., Brooklyn, N. Y.

#### MASSAGE CREAM

White Beeswax	20	Water	45
Spermaceti	20	Borax	3
Peanut Oil	45	Camphor	1
Lanolin	12		

Melt the wax over a water bath, add the lanolin and oil, and heat to 80°C. The camphor is dissolved in the oil with gentle heat, the borax is dissolved in the water and heated to 80°C. At this temperature the water is added to the wax oil mixture at the same temperature with vigorous stirring; best done with a motor stirrer.<sup>21</sup>

# SPORTS CREAM

Glycosterin	12	Cetyl Alcohol	3
Mineral Oil	10	Water	100

Heat to 60°C. and mix vigorously till emulsified.22

# NAIL CREAM

a.	Beeswax, White	15.0	Borax	1.5
	Ozokerite, White	2.5	c. Aluminum Stearate	20.0
	Montan Wax, Bleached	2.5	d. Fish Silver Paste (10%	
	Mineral Oil	60.0	Dispersed in Amyl	
	Cetyl Alcohol	2.0	Acetate)	5.0 g.
b.	Water	23.5	·	_

Melt a, add b of about 70°C. Stir thoroughly, add c. Stir until the emulsion is almost cold, add d.<sup>23</sup>

# MOSQUITO REPELLING CREAM

# FORMULA No. 1.

a. Wheat Starch	5	c. Lanolin	30
Water	10	d. Clove Oil	5-10
b. Glycerin (28° Bé.)	45		

Grind a until homogeneous, add b, and warm gently until a homogeneous jelly is formed. Cool, and grind now with c

<sup>&</sup>lt;sup>21</sup> Ibid., Vol. 2, p. 149 (1935).

<sup>&</sup>lt;sup>22</sup> Ibid., Vol. 4, p. 53 (1939).

<sup>&</sup>lt;sup>23</sup> Ibid., p. 67.

and d in a mortar very thoroughly until distribution is satisfactory. Fill at once into collapsible tubes.<sup>1</sup>

	FORMUL	A No. 2.	
Glyceryl Monostearate Oil of Cedar Leaf Oil of Pennyroyal Linalyl Acetate Gasoline	11.0 4.0 4.0 3.0 5.0	Menthol Phenol Glycerin Water	0.5 2.0 5.0 65.5

Put the glyceryl monostearate into the water, add the glycerin and bring the mixture to the boiling point with constant stirring. Keep stirring and when the temperature drops to about 45°C. add the rest of the ingredients which have been mixed together.<sup>2</sup>

	FORMUL	A No. 3.	
White Mineral Oil	4 oz.	Distilled Water	8 oz.
Beeswax U.S.P.		Borax	30 gr.
Spermaceti		Butyl Salicylate <sup>3</sup>	1.5 oz.

# ANTISEPTIC CREAM

# FORMULA No. 1.

Mixed Mono- and Di- Fatty Esters of Polythylene Glycol Stearic Acid Paraffin Wax		Mineral Oil Petrolatum Oxy-Quinoline Water <sup>4</sup>	 make	15 10 0.2 100	
Fo	RM ULA	No. 2			

# FORMULA No. 2.

Sulphonated Castor Oil Methyl Salicylate Eucalyptol Menthol	150 150 45 5	Potassium ( Glycerin Water <sup>5</sup>	Carbonate	50 100 500
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# ACRIFLAVINE EMULSION

Acriflavine		Distilled Water	25 cc.
Wool Fat		Liquid Paraffin	925 cc.
	•	7	720 CC.

<sup>&</sup>lt;sup>1</sup> Bennett, Chem. Formulary, Vol. 3, p. 68 (1936).

<sup>&</sup>lt;sup>2</sup> Ibid., Vol. 2, p. 363 (1935). <sup>3</sup> Ibid., Vol. 1, p. 111 (1933).

<sup>&</sup>lt;sup>4</sup> Goldschmidt, U. S. 2,269,529 (1942).

<sup>&</sup>lt;sup>5</sup> Bennett, Chem. Formulary, Vol. 2, p. 157 (1935).

Dissolve the acriflavine in the distilled water, heated to about 90°, and add the solution to the anhydrous lanolin in a large warmed mortar. Triturate until a thick cream is produced, then add gradually the liquid paraffin.<sup>6</sup>

#### WHITE OIL EMULSION

Agar	0.7	Sodium Benzoate	0.2
Gum Acacia	2.1	Flavoring	0.7
Tragacanth	0.3	Water 7	46
White Mineral Oil	50.0		

#### SUN-BURN PREVENTIVE PREPARATIONS

#### FORMULA NO. 1.

Quinine Sulphate	3	Lanolin, Anhydrous	15.0
White Ceresin Wax	5.5	Water	35.5
White Petrolatum	20.5	Cassia Oil	1.0
Mineral Oil	19.5		

Heat the water to 70°C. and dissolve the quinine sulphate in it. Melt the ceresin, petrolatum, and lanolin together, stir in the mineral oil, bring the mixture to 65°C. and stir in the quinine solution. Continue stirring until the temperature drops to 45°C. and then add the cassia oil.<sup>1</sup>

### FORMULA No. 2.

Quinine Bisulphate	2.5	Alcohol	10.0
Cholesterin Absorption Base	25.5	Water	49.5
\umeral Oil	12.5	Perfume <sup>2</sup>	.5

### FORMULA No. 3.

a. Triethanolamine	40	Trihydroxyethylamine	
		Stearate	40

### Melt on water bath, make emulsion in

Water (60°C.)	620-630	Peanut Oil	150
b. Paraffin Oil	100	Oleic Acid	30

<sup>6</sup> Ibid., p. 371.

<sup>7</sup> Ibid.

<sup>&</sup>lt;sup>1</sup> Ibid., p. 150.

<sup>&</sup>lt;sup>2</sup> Thid.

Warm up on water bath to 40°C.

Methyl-p-Hydroxy Benzoate 1

Pour b into a, perfume with

c. Perfume Oil

to suit

Stir until cold.3

### SUNBURN HEALING PREPARATIONS

### FORMULA No. 1. (Healing Oil)

Tincture of Benzoin	Olive Oil	40
Borax	Lime Water	52

Dissolve the borax in the lime water; add the olive oil with rapid agitation. When an emulsion is formed add the tincture of benzoin.<sup>4</sup>

#### HAIR CREAMS

#### FORMULA NO. 1.

Cholesterin	2	White Mineral Oil	50
White Petrolatum	10	Water	100
Lanolin	10		

The lanolin may be replaced by deodorized arachis oil and beeswax (1:5). It is necessary to use a homogenizer in the preparation of such a cream.<sup>1</sup>

#### FORMULA No. 2.

Gum Tragacanth (pulv.)		Sodium Benzoate	0.2
Castor Oil		Formaldehyde	0.4
Glucose	8.0	Water to	160.0
Alcohol	14.0	Perfume 2	Sufficient

<sup>&</sup>lt;sup>3</sup> Bennett, Chem. Formulary, Vol. 3, p. 98 (1936).

<sup>&</sup>lt;sup>4</sup> Ibid., Vol. 2, p. 153 (1935).

<sup>&</sup>lt;sup>1</sup> Ibid., Vol. 4, p. 66 (1939). <sup>2</sup> Ibid., Vol. 2, p. 161 (1935).

#### FORMULA No. 3.

Gum Tragacanth (pulv.)	1.5	Formaldehyde	0.4
Castor Oil	4.0	Water to	160.0
Glycerin	8.0	Perfume	Sufficient
Alcohol	14.0	Mineral Oil 3	48.0
Sodium Benzoate	0.2		

Formula No. 4.			
Powdered Soap Water	50 36	Glycerin	18

Dissolve the soap in hot water, add the glycerin. While stirring well add

Peanut Oil

40

Mix until homogeneous and add

Perfume

12

and continue stirring until nearly cool. To get maximum stability run through a colloid mill.4

#### HAIR MILK

#### FORMULA No. 1.

Glyceryl Monostearate	Distilled Water	64
Liquid Paraffin	Perfume <sup>5</sup>	To suit

#### FORMULA No. 2.

1. Mineral Oil, White	144	3. Water, Warm	320
2. Trihydroxyethylamine		4. Perfume	3
Stearate	29		

While stirring heat (1) and (2) until melted together. Add (3) slowly with stirring until uniform. Add (4). Stir. Allow to stand overnight, stir moderately and package.

<sup>3</sup> Ibid.

<sup>&</sup>lt;sup>4</sup> Bennett, Chem. Formulary, Vol. 4, p. 66 (1939).

<sup>&</sup>lt;sup>5</sup> Ibid., Vol. 4, p. 63.

<sup>6</sup> Ibid., Vol. 1, p. 147 (1933).

#### FORMULA No. 3.

Triethanolamine Stearate	7	Distilled Water	65
Spermaceti	3	Perfume	to suit
Liquid Paraffin	25		

Dissolve the trihydroxyethylamine stearate and spermaceti in the liquid paraffin over a water-bath. While still warm run in the water previously warmed. Stir vigorously until quite homogeneous and cool, working in the perfume as the cream cools.<sup>7</sup>

#### MILKY HAIR WASH

1. Trihydroxyethylamine		3. Pine Oil	6 1Ь
Stearate Special	10 lb.	4. Water	250 lb
2. Kerosene	150 lb.		

Heat Nos. 1 and 2 to 140°F. and stir until dissolved; then stir in No. 3. Now allow No. 4 to run in slowly while stirring. If the pine oil is objectionable, however, any other oil may be substituted for it. It may be colored by means of any water-soluble dye free from salt.8

#### PINE TAR SHAMPOO

Pine Tar	25	Industrial Spirit	200
Yellow Soft Soap	300	Water 1	475

#### SOAPLESS SHAMPOO

Sulfonated Olive Oil, concen-		White Mineral Oil	15
trated	40	Water	35
Sulfonated Castor Oil, con-		25% Solution of Caustic Soda	
centrated	10	to Clear	

Mix all the ingredients with the exception of the caustic soda, warm to 45–50°C. and add enough of the caustic soda solution (1 or 2%) until the mixture turns bright. Perfume as desired.<sup>2</sup>

<sup>&</sup>lt;sup>7</sup> Ibid., Vol. 4, p. 63 (1939).

<sup>8</sup> Ibid., Vol. 1, p. 119 (1933).

<sup>&</sup>lt;sup>1</sup> Bennett, Chem. Formulary, Vol. 2, p. 159 (1935).
<sup>2</sup> Ibid., Vol. 1, p. 119 (1933).

#### HAIR TONIC

Cholesterol	 Sodium Choleate	5
Mineral Oil	Glycerin	50
Heat to 130°F		

Heat to 130° and add to above slowly with agitation. Then add slowly with agitation a solution of

Borax 5 Water 1000

That has been heated to 130°F. Stir until cold.3

#### PINE OIL EMULSIONS

#### FORMULA No. 1.

1. Pine Needle Oil	10	3. Water	5
2. Sodium Sulforicinoleate	10	<ol><li>Fluorescein</li></ol>	To Suit

Mix 1 and 2 until dissolved. Add 3 slowly with stirring. Add 4 and stir until dissolved.<sup>1</sup>

#### FORMULA No. 2.

Turkey Red Oil	10	Pine Oil
Fluorescein		Water

Dissolve the fluorescein in the turkey red oil; add the pine oil and when well mixed add the water, stirring until a uniform liquid results. Strain if necessary.<sup>2</sup>

#### FORMULA No. 3.

Cocoanut Oil (Saponification	Pine Oil	40.0
No. 257) 160.0	Water	754.0
Potassium Hydroxide (89%) 46.0		

Cocoanut oil of Ceylon Grade is heated to a temperature of 80°-85°C. The potassium hydroxide is then dissolved in a sufficient amount of the water to make a 15% to 20% solution. One-half the solution is then added to the cocoanut

<sup>2</sup> Ibid., p. 101.

<sup>&</sup>lt;sup>3</sup> Ibid., Vol. 2, p. 163 (1935).

<sup>&</sup>lt;sup>1</sup>Bennett, Chem. Formulary, Vol. 1, p. 102 (1933)

oil and stirred in slowly. The balance of water is then added followed by the balance of potassium hydroxide solution which is stirred in slowly. The temperature of the mix is then kept at 80°-85°C. for a period of from two to three hours with good agitation. After complete saponification, the solution is then cooled, chilled and filtered in this chilled state. The Pine Oil is then added by stirring in very slowly. A sufficient amount of water is then added to balance water loss during sustained heating to bring product to original weight.<sup>3</sup>

Mixed Oleic Acid Esters of		Diglycol Monooleate	7
Polyglycol	10	Oxalic Acid	2
Pine Oil	40	Water 3a	41

#### SOLIDIFIED PERFUME (OILS)

Trihydroxyethyl Amine	Orange or Other Oil
Linoleate	Water

Add in above order stirring well.4

# WATER SOLUBLE PERFUMES (Jellified Perfumes)

1.	Trihydroxyethylamine
	Linoleate #297

2. Perfume3. Water

Mix Nos. 1 and 2 together until uniform. Add No. 3 slowly with stirring until a jelly is formed. The water must be added VERY slowly, stirring thoroughly, but as soon as a turbidity appears no more water can be added. These jelly perfumes disperse in water to give a milky solution when concentrated and a slightly turbid solution when highly diluted. By incorporating medicinal ingredients, ointments, salves, etc., are made which are not sticky and wash off readily with water.<sup>5</sup>

<sup>3</sup> P. C.

<sup>&</sup>lt;sup>3a</sup> Goldschmidt, U. S. 2,269,529 (1942).

<sup>4</sup> Bennett, Chem. Formulary, Vol. 1, p. 126 (1933).

<sup>&</sup>lt;sup>5</sup> Ibid., p. 127.

	FORMUL	A No. 3.	
Glycosterin Ethylene Glycol Mineral Oil White	10 10 8	Lanolin Stearic Acid Glycerin <sup>3</sup>	2 34 2
	FORMUL	A No. 4.	
<ol> <li>Mineral Oil</li> <li>Glycosterin</li> </ol>	10 10	3. Water	50

Heat (1) and (2) to 150°F. and stir (3) into it heated to 150°F. slowly. A little perfume and menthol (if desired) is stirred in at 105°F. and stirring is continued until cold.4

	Formula	No. 5.	
Stearic Acid White Mineral Oil Paraffin Wax	12 12 5	Soap Flakes Water <sup>5</sup>	3 72
	FORMULA	No. 6.	
Stearic Acid Lanolin Raisin Seed Oil	16.5 1.0 8.5	Triethanolamine Water Perfume <sup>6</sup>	1.0 72.5 0.5
	FORMULA	No. 7.	
Glyceryl Monostearate Stearic Acid Mineral Oil	6.5 6.5 4.0	Peanut Oil Glycerin Water <sup>7</sup>	4.0 10.0 69.0
	SHAVING	CREAMS	

#### FORMULA No. 1.

		2.5 g.
		39.0 g.
		2.0 g.
	Cetyl Alcohol	1.0 g.
8.0 g.	Menthol Crystals	0.22 g.
	8.0 g. 3.5 g. 1.5 g.	3.5 g. Water 1.5 g. Olive Oil

In preparing this cream, the stearic acid and oils are mixed and heated to 212°F. At this point the alkali, likewise heated

<sup>8</sup> Bennett, Chem. Formulary, Vol. 1, p. 131 (1933).

<sup>4</sup> Ibid.

<sup>&</sup>lt;sup>5</sup> Ibid., p. 129.

<sup>6</sup> Ibid., Vol. 4, p. 73 (1939). 7 Ibid., Vol. 1, p. 129 (1933).

to the same temperature, is slowly added with stirring. The mixture is then held at 210°F. for 16 hours and stirred every 3 or 4 hours during this time. Next, the boric acid dissolved in half the water content is added at 212°F, and mixed. Finally, the remaining constituents mixed with water at 212°F, are added and the composition is stirred slowly while cooling.1

### FORMULA No. 2.

Glyceryl Monostearate	6.5	Peanut Oil	4.0
Stearic Acid	6.5	Glycerin	10.0
Mineral Oil	4.0	Water 2	69.0

#### DISINFECTANT EMULSIONS

#### FORMULA No. 1.

100 Caustic Soda (36° Bé.) 51.2 Sulphonated Castor Oil

Heat above at 80-100°C., then add

Rosin 104

Mix with heating until uniform and add

Tar Oils (200-320°C.) 775

Mix until dissolved and then add

Water 1 To make 1000

#### FORMULA NO. 2.

Cresol	35	Castor Oil Soap	20
Creosote	45	Water 2	To Suit

#### FORMULA NO. 3.

Rosin	17	Caustic Soda	2
Washed Cresote	71	Water <sup>3</sup>	2
Cresol	10		

<sup>&</sup>lt;sup>1</sup> Atlas Powder Co., Wilmington, Del.

Bennett, Chem. Formulary, Vol. 1, p. 129 (1933).
 Bennett, Chem. Formulary, Vol. 3, p. 103 (1936).

<sup>&</sup>lt;sup>2</sup> Ibid., Vol. 1, p. 150 (1933).

<sup>8</sup> Ibid.

#### FORMULA No. 4.

Cresol	50.0	Castor Oil Soap	0.5
Cresote	7.5	Caustic Soda	0.1
Glue	2.5	Water 4	60.0

#### FORMULA NO. 5.

Cresol	50	cc.	Potassium Hydroxide	4.2	g.
Linseed Oil	17	g.	Distilled Water, To	100	cc.
Oleic Acid	1	g.			

The oil and acid are heated to the maximum temperature of the water-bath and a solution of the potassium hydroxide in 25 mils. of water, heated nearly to boiling, is added.5

#### FORMULA NO. 6.

A very satisfactory water-soluble disinfectant is a mixture of equal parts of cresol and Turkey red oil.6

#### FORMULA No. 7.

Cresylic Acid	100	Caustic Potash Solution	
Linseed Oil	100	(331/3%)	75
		Alcohol 7	6

#### FORMULA No. 8.

Tar Oil Acids	40	Caustic Potash Solution	
Rosin	10	(331/3%)	70
		Alcohol 8	70

#### FORMULA No. 9.

Cresylic Acid or Cresol	50	Sodium or Potassium Hydroxide 1
Sulfonated Castor Oil (50%)	24	Water 18
Red Oil or Oleic Acid	7	

Mix the red oil and cresol warm. Dissolve the hydroxide in the water, warming if necessary. Mix these two solutions and add the sulfonated castor.9

<sup>4</sup> Ibid.

<sup>&</sup>lt;sup>5</sup> Ibid., Vol. 2, p. 273 (1935). <sup>6</sup> Ibid., Vol. 4, p. 91 (1939).

<sup>7</sup> Ibid.

<sup>&</sup>lt;sup>8</sup> Bennett, Chem. Formulary, Vol. 4, p. 91 (1939).

<sup>9</sup> Ibid., Vol. 2, p. 273 (1935).

Caustic Soda (25% Solution) 10.30

FORMULA I	No. 1	0.
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66.00

20.00

FORMULA NO. 11.				
Light Coal Tar Oil (Sp. Gr. about 1.02 at 60°F.)	69.4	Caustic Soda Solution (Sp. Gr. 1.3)	9.6	
Rosin (Grade F)	18.4	Water	2.6	

Water 10

This formula is stated in percentages by weight.

Melt rosin and add oil, mix well. Add soda and mix. Add water.<sup>11</sup>

#### FORMULA No. 12.

Tar Acids	<b>7</b> 5	Mineral Oil	4
Oil Soluble Mineral Oil		Isopropyl Alcohol 12	10
Sulphonate	11		

#### PINE OIL DISINFECTANT

#### FORMULA No. 1.

Pine Oil Naphthenic Soap	60 25	Sulphonated Castor Oil 1	15
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#### FORMULA No. 2.

Pine Oil	57.00	Glucose	1.00
Rosin	25.00	Water	8.50
Caustic Potash (25%			
Solution)	8.50	•	

The caustic potash and water are mixed and heated. When boiling the rosin is added slowly and the heating is continued for one hour taking care to stir mixture occasionally, then the pine oil is added and the heating is continued until a sample that has been withdrawn and poured into water does not show separation of oil. When the test is satisfactory the heat is removed and the glucose is added.

For use add one ounce to gallon of water.2

Tar Acid Oil

Rosin

<sup>10</sup> Ibid.

<sup>11</sup> Ibid.

<sup>&</sup>lt;sup>12</sup> Schuler, U. S. 2,228,407 (1941).

<sup>&</sup>lt;sup>1</sup> Bennett, Chem. Formulary, Vol. 4, p. 91 (1939).

<sup>&</sup>lt;sup>2</sup> Ibid., Vol. 2, p. 273 (1935).

FORMULA	* T -	~
HODINITI	NO	

Pine Oil		Water 8	8 lb.
Soap	29 lb.		

#### FORMULA NO. 4.

Raw Pine Oil	60	Red Oil or Oleic Acid	9
Sulfonated Castor Oil (50%)	30	Potassium Hydroxide (Solid)	1

Mix the pine oil and the sulfonated castor together. Then add the red oil and dissolve the potassium hydroxide in the mixed oils.4

### FORMULA NO. 5.

Rosin Soap 10 lb. Pin	e Oil 60 1b	ı.
-----------------------	-------------	----

The pine oil is worked into the soap gradually, to avoid lumping. Part of the pine oil may be replaced by kerosene to lower costs. The above when stirred into water gives a milky white emulsion.5

#### FORMULA No. 6.

Pheno1	1 oz.	Sulphonated Oliv	e Oil
Pine Oil	5 oz.	(30%)	16 oz.
		Water 6	78 oz.

#### FORMULA No. 7.

Oleic Acid (Acid Number		Sodium Hydroxide (100%)	16.3
194)	61.6	Yarmor Pine Oil	133.0
"1" Wood Rosin (Acid		Tri-sodium Phosphate	26.7
Number 165)	61.6	Water 7	700.8

#### ANTISEPTIC EMULSION

#### FORMULA NO. 1.

Oil Soluble Mineral Oil		Eucalyptol	5
Sulphonate	11	Isopropyl Alcohol	20
Thymol	5	Kerosene 8	59

<sup>&</sup>lt;sup>3</sup> Ibid., Vol. 4, p. 91 (1939).

<sup>&</sup>lt;sup>4</sup> Ibid., Vol. 2, p. 273 (1935). <sup>5</sup> Ibid., Vol. 1, p. 150 (1933). <sup>6</sup> Ibid., Vol. 4, p. 92 (1939).

<sup>&</sup>lt;sup>7</sup> Hercules Powder Co., Wilmington, Del.

<sup>&</sup>lt;sup>8</sup> Schuler, U. S. 2,228,407 (1941).

	Cosmetic and I	Orug Emulsions	293
	FORMUL	A No. 2.	
Cetyl Alcohol Medicinal Soap Glycerin	68 18 23	Mineral Oil Water Perfume <sup>9</sup>	91 780 20

<sup>9</sup> Bennett, Chem. Formulary, Vol. 4, p. 59 (1939).

#### CHAPTER VII

### DEFOAMING AGENTS

### Defoaming

Octyl alcohol is used as a defoaming agent as follows (all suggested concentrations are by volume unless otherwise indicated):

- (a) Oil, where foaming is due to presence of water, use 0.0005%.
- (b) Varnish inks, use 0.001-0.01%.
- (c) Beet sugar, use 0.0005-0.005% of solution.
- (d) Paper, use 0.0005-0.005% of pulp.
- (e) Analytical procedure; such as analyses of fruit juices, powdered milk, petroleum-water mixtures, etc., use a few drops in 500 cc.
- (f) Photographic film developing solutions. Spray 1 to 10 oz. for 200 gal. of foam.
- (g) Textile printing, use 0.25-1.5% of paste.
- (h) Chemical Processes (manufacture dyes, alum, etc.) use .1-1.0%.
- (i) Wire drawing soap lubricants, use 0.01-0.1%.
- (j) Asphalt emulsions, use 0.001-0.01%.
- (k) Eliminating air bubbles and "pin holes" in gelatin glue spreading, use 0.1-1.0%.

Where the odor of octyl alcohol is objectionable the proprietary de-foamer "Foamex" is used similarly to the above.

<sup>&</sup>lt;sup>1</sup> Bennett, Chem. Formulary, Vol. 4, p. 100 (1939).

#### DEFOAMING AND DEFROTHING AGENTS

#### FORMULA NO. 1.

5 parts of aluminum stearate is dissolved in 95 parts of pine oil, to give a substantially clear fluid solution.<sup>1a</sup>

#### FORMULA No. 2.

5 parts of aluminum stearate is dissolved in a mixture of 78.75 parts of pine or eucalyptus oil and 26.25 parts of a mixture of higher alcohols obtainable as a by-product during the manufacture of methanol.

The addition of one per cent or less of either of the above to a solution of 10 parts of glue and 10 parts of dextrine in 100 parts of water completely prevents the development of foam or froth when the mixture is shaken."

#### FORMULA No. 3.

5 parts by weight of aluminum stearate is dissolved in 95 parts of the fraction boiling from 195°C. and up of the liquid oxygen-containing organic compounds obtainable as a by-product in the known synthesis of methanol under pressure from hydrogen and oxide of carbon.

The addition of 1 per cent or less of the above solution to a solution consisting of 20% of animal glue in water completely prevents the development of foam or froth when the mixture is shaken.<sup>3</sup>

#### FORMULA No. 4.

5 parts of zinc stearate is dissolved in 95 parts of pine oil at 95–100°C.4

<sup>&</sup>lt;sup>1a</sup> Bennett, Chem. Formulary, Vol. 2, p. 183 (1935).

<sup>&</sup>lt;sup>2</sup> Ibid.

<sup>&</sup>lt;sup>3</sup> Ibid.

<sup>4</sup> Ibid.

#### FORMULA NO. 5.

3 parts of calcium oleate is dissolved in 97 parts of pine oil at 95–100°C.4a

The addition of 0.25% of either of the above two agents to a solution of 10 parts of glue and 10 parts of dextrine in 100 parts of water considerably reduces the amount of foam formed when the mixture is shaken as compared with the amount normally formed.<sup>5</sup>

When 0.25% of pine oil alone is added, the amount of froth formed is also sensibly reduced but still remains about double that formed according to the above process.

#### FORMULA No. 6.

5 parts of barium stearate is dissolved in 95 parts of oleic acid at 95–100°C.

The addition of ½ % of this agent to glue-dextrine solutions reduces the froth formed on shaking to half the amount formed when ½ % of oleic acid alone has been added.6

#### RADIATOR ANTI-FOAMING AGENT

Water	1000	Potassium Hydroxide 7	12
Montan Wax	8		

<sup>4</sup>a Ibid.

<sup>5</sup> Ibid.

<sup>#</sup> TL 1

<sup>&</sup>lt;sup>7</sup> Flaxman, U. S. 2,127,490 (1938).

### CHAPTER VIII

## FOOD EMULSIONS

n	т	77	$\Gamma$ T	77	n	c	$\sim$	$\sim$	~	~	TΥ	٠ ٣	$\sim$	٦.	۲.	$\sim$

### Boil to 245°F.

Brown Sugar	8 1b.	Water	4 pt.
Butter	4 lb.	Salt	2 oz.

### Now cream up lightly

Powdered Sugar	25 lb.	Liquid Skimmed Milk	2 pt.
Shortening	5 lb.		

Into this creamed mass add the hot butterscotch syrup and whip up to the desired consistency 1

### CHOCOLATE MILK, STABILIZED

Sodium Alginate	0.8 1b.	Cocoa	8.8 1b.
Irish Moss	0.7 1b.	Milk 8	96.0 gal.
Sugar	44.0 lb.		•

#### COCOA ICING

## Beat together until smooth and glossy:

Plastic Cocoanut Butter	16	Icing Sugar	88
Invert Sugar	20	Milk Powder	43/4
Water	12	Salt	1/8
Cocoa	20	Vanilla 2	• •

<sup>Fiene and Blumenthal, Handbook of Food Mfg. (1938).
Bennett, Chem. Formulary, Vol. 1, p. 54 (1933).</sup> 

#### ARTIFICIAL CREAM

#### FORMULA NO. 1.

Butter, unsalted	2 lb.	Sugar	oz.
Milk	2 pt.	Sodium Alginate	, oz.

Warm the milk and butter together to 100°F., add the sugar and alginate and stir thoroughly until dissolved. Homogenize without the addition of further heat and without allowing the mixture to cool. If the cream is to be kept for any length of time the mixture should be pasteurized at 140°F. for half an hour before homogenizing and then passed through a sterilized homogenizer.<sup>3</sup>

	FORMUL	A No. 2.	
Butter Fat	19.00	Gelatin	1.00
Vegetable Fat	10.00	Borax	0.25
Milk Powder	7.00	Water	62.00
Sugar	0.75	Flavor 4	To suit

#### FORMULA No. 3.

To produce a 32% fat synthetic shipping cream, use 1 lb. 10 oz. of emulsifying type shortening, 2 lb. 15 oz. water, 8 oz. dry milk solids and not over  $1\frac{1}{2}$ % fat.

To produce a half and half cream made from half uncultured butter and half emulsifying type shortening, use 1 lb. 14 oz. uncultured butter, 1 lb. 10 oz. emulsifying type shortening, 1 lb. dry milk solids not over  $1\frac{1}{2}\%$  fat, 5 lb. 9 oz. fresh water.<sup>5</sup>

### SYNTHETIC "CREAM" FOR CONFECTIONERY

As an example, a whale oil is used which has been hydrogenated until its melting point is about 30°C. (86°F.) A

<sup>3</sup> Ibid., Vol. 4, p. 131 (1939).

<sup>&</sup>lt;sup>4</sup> Ibid., Vol. 1, p. 57 (1933).

<sup>&</sup>lt;sup>5</sup> Ibid., Vol. 5, p. 145 (1941).

15

blend is made from 10 lb. of this hardened fat, 5 lb. of buttermilk, 1.5 lb. sugar and 12 oz. of glycerin. The resulting emulsion is stirred thoroughly while passing in air, to form a creamy product.<sup>6</sup>

### CHEESE, ICE-CREAM AND SALAD STABILIZER

Locust Bean Gum 65 Karaya Gum

Irish Moss. Powdered 35

When used in the preparation of cream cheese, the undiluted mixture of the three ingredients mentioned above is added at the time that the curds are mixed with the cream in the usual procedure for the manufacture of cream cheese and in the proportion of about one-half of 1% by weight on a wet basis. The material is heated to about 165°F., homogenized, and then packed hot.

In ice cream it is used diluted with sugar, in the preferred proportion of one-half of 1% on a wet basis, the stabilizer acts to prevent crystallization of ice particles and thus insures a fine, smooth texture and a body which will hold up under severe shocks.<sup>7</sup>

### DAIRY PRODUCT AND STABILIZER

Select Irish Moss (Pulverized) Ordinary Irish Moss	20 1ь.	Glyceryl Mono Stearate Tri Sodium Phosphate	9 lb. 8 oz.
(Pulverized)	20 1ь.		

The process of making the stabilizer is as follows:

The two grades of Irish moss, the glyceryl mono stearate, and the tri-sodium phosphate with the addition of 10 times their weight of water are heated in a mixing kettle. Preferably, the heating is done by direct steam injection which adds to the moisture content and also serves as an agitator to

<sup>6</sup> Ibid., p. 145-6.

<sup>&</sup>lt;sup>7</sup> Ibid., Vol. 3, p. 150 (1936).

<sup>8</sup> Green, U. S. 2,097,225 (1937).

insure complete admixture of the ingredients. The temperature is brought to the boiling point and held at that degree for approximately 30 minutes during which time the steam circulates through the batch, the odor of the Irish moss being carried off in the escaping steam.

The batch is then pumped to an atmospheric double drum drier or its equivalent, and all but approximately 1% to 6% of its moisture content is removed. Where a drier of the type described is employed, the moisture is, of course, removed by the heated rolls. The material comes off the drier in flake form, and it is then pulverized which makes it ready to be incorporated in the dairy powder from which the chocolate milk and other dairy products are made.<sup>9</sup>

#### INFANTS' MILK SYNTHETIC

Sugar	40	Dextrin	20
Soya Bean Powder	125	Egg Yolk, Liquid	50
Lactose	30	Calcium Lactate	6
Peanut Oil	20	Salt	2

Stir in water before use.10

#### LEMON OIL EMULSIONS

FORMULA No. 1.

Gum Tragacanth 18 oz. Water 4 gal.

Place the gum tragacanth in a kettle similar to a baker's mixing machine. Pour the water over it, stir well and whip until all the lumps have disappeared. Let stand overnight.

Next morning squeeze the gum solution through a cheesecloth bag. Add to the homogeneous gum solution

Glycerin 1 qt.

Place the kettle under the machine and allow to mix for 5 minutes at slow speed.

Bennett, Chem. Formulary, Vol. 5, p. 146 (1941).
 Bennett, Chem. Formulary, Vol. 3, p. 151 (1936).

Now put the machine in second speed and gradually add

Lemon Oil 4 qt.

After 1 quart of oil has been added, it is advisable to put the machine in third speed, as, otherwise, the oil will not be taken up by the gum solution. When all the oil has been added, stop the machine, scrape the walls of the kettle with a long-bladed spatula and add 5 ounces of benzoate of soda in solution, to prevent fermentation. Start the machine again in third speed for about 5–10 minutes.<sup>1</sup>

#### FORMULA No. 2.

1. Gum Arabic	13 oz.	3. Lemon Oil	20 oz.
2. Terpeneless Oil of		4. Glycerin	40 oz.
Lemon	20 oz.	5. Water	To make 10 gal.

Mix 1 and 4, then mix in 2 and 3 to this; add 5 slowly with good stirring. Beat intermittently until homogeneous. Then pass through an homogenizer.<sup>2</sup>

#### TRANSPARENT LEMON OIL EMULSION

Invert Sugar Syrum (80%) 60 Medium Fine Granulated	lb. Water 16 lb. Gelatin (180 Bloom-Min.) 4 oz.
Sugar 24	1b.

The gelatin is dissolved in water, corn syrup added and the mixture heated until solution is complete. The invert syrup is then added and the whole thoroughly mixed. The refractive index of this solution should be approximately 1.4720. In the preparation of the emulsion, 20 ounces of terpeneless and 20 ounces of regular lemon oil are mixed with enough of the above solution to produce 10 gallons. This mixture is then homogenized and left standing for 18 hours which allows entrapped air bubbles to rise to the surface. Glass containers are filled from the bottom outlet of

<sup>2</sup> Ibid.

<sup>&</sup>lt;sup>1</sup> Fiene and Blumenthal, Handbook of Food Mfg. (1938).

the emulsion container and tightly stoppered. The resultant emulsion will be practically transparent.<sup>3</sup>

#### ORANGE OIL EMULSION

Gelatin	4 oz.	Invert Sugar	60 lb.
Water		Terpeneless Orange Oil	20 oz.
Cane Sugar	24 lb.	Orange Oil	20 oz.

Dissolve the gelatin in the water, add the cane sugar and heat until dissolved. Then add the invert sugar and mix well; homogenize.\*

### ORANGE EMULSION (CLOUDY)

Gum Arabic (Best Grade) 1 lb. Water

1 pt.

Let stand overnight; mix and filter. Add

Orange Oil

6 dr.

Mix and pass through a colloid mill. Use  $\frac{1}{2}$  oz. of above per gallon of syrup.<sup>5</sup>

#### BITTER ALMOND OIL EMULSION

Gum Arabic Bitter Almond Oil 10 Cane Sugar 2 Water

120

Mix together the sugar and gum. Then add it to the water, which is agitated by means of a rapid mixer, then allow to mix until a uniform emulsion results. Then add the oil slowly until completely emulsified.<sup>6</sup>

#### MARGARINE

### FORMULA No. 1.

1. Glycerol Monostearate 85 lb. 3. Cotton Seed Oil, 2. Coconut Oil (76°F. Winterized 125 lb. Titer) 800 lb. 4. Cultured Milk 50-55 gal.

6 P. C.

<sup>&</sup>lt;sup>3</sup> Bennett, Chem. Formulary, Vol. 5, p. 119 (1939).

<sup>4</sup> Handbook of Food Mfg., Fiene and Blumenthal.

<sup>&</sup>lt;sup>5</sup> Bennett, Chem. Formulary, Vol. 4, p. 45 (1939).

Melt 1, 2 and 3 and mix; churn with milk, temper, work and salt.<sup>1</sup>

FORMULA No. 2.

Soya Bean Lecithiu (60-65%)

Glyceryl Distearate

100 lb.

Melt together and mix in

Water Hot

100 1ь.

100 lb.

Add and mix in

Water Warm

500 1b.

One lb. of above is added to 100 lb. plastic margarine.<sup>2</sup>

#### FORMULA No. 3.

<ol> <li>Water</li> <li>Galagum C</li> </ol>	120	4. Caustic Soda	0.02
	1	5. Butter Flavor	To suit
3. Cottonseed Oil	40	J. Butter Fravor	10 suit

Dissolve 4 in 1 and strew 2 on surface; bring to a boil while stirring; run 3 and 5 into it slowly with high speed intermittent stirring.<sup>8</sup>

### MAYONNAISE AND SALAD DRESSINGS

#### MAYONNAISE

#### FORMULA No. 1.

Frozen Egg Yolk 120 lb. Spice Mixture\*
Winter Oil (Cottonseed) 330 lb. Salt 200 lb.
Spice Mixtures\* 24 lb. Mustard (Ground) #1 100 lb.
Vinegar \*\* 160 lb.

\* Refer to government definitions and standards for food products.

\*\* Vinegar: Use cider vinegar. Dilute 9 gallons of vinegar with 1 gallon of

Mix the frozen yolk, which must be taken out of the refrigerator the night before making the batch, with a part of the vinegar and the spices. Be sure to keep some vinegar back

<sup>&</sup>lt;sup>1</sup> Bennett, Chem. Formulary, Vol. 5, p. 146 (1941).

<sup>&</sup>lt;sup>2</sup> Ibid.

<sup>&</sup>lt;sup>3</sup> Ibid., Vol. 1, p. 47 (1933).

Vinegar (50 grain)

Water

to be added last or if the batch breaks. Allow the oil to flow in slowly.<sup>1</sup>

### FORMULA No. 2.

## Thicker than Formula No. 1. Milder in taste.

Frozen Egg Yolk Winter Oil (Cottonseed Oil)	7½ 1b. 33 1b.	Spice Mixture (As in Formula #1) Vinegar (As in Formula #1) <sup>2</sup>	2 lb. 10 lb.
	Formul	A No. 3.	
Cotton Seed Salad Oil Egg Yolk	70.25 lb. 10.00 lb.	Salt Sugar	1.45 lb. 3.50 lb.

The egg yolk can be reduced to 7.5 lbs. The sugar can be left out.<sup>3</sup>

10.00 lb. Mustard

3.90 lb. White Pepper

.80 1ь.

.10 lb.

#### FORMULA No. 4.

Frozen Egg Yolk	4½ lb.	Mustard #2	8 oz.
Winter Pressed Cotton	ıseed	White Vinegar	1 qt.
Oil	75 lb.	Water 4	2 qt.
Salt	13 oz.		_

#### FORMULA No. 5.

Egg Yolk (Fresh)	12 lb.	Paprika	¾ oz.
Sugar	2⅓ lb.	Pepper (White)	3/4 oz.
Mustard Flour	1 1b.	Oil (Salad)	13 gal.
Salt	1 lb. 7 oz.	Vinegar (100 grain, white)	4 qt.
Onion Juice (Fresh)	2 oz.	Water	2 qt.

The yolks are thrown into the beater bowl. Set the beater at high speed. Beat the yolks until they are well broken up which requires about a minute.

<sup>&</sup>lt;sup>1</sup> Fiene and Blumenthal, Handbook of Food Mfg. (1938).

<sup>&</sup>lt;sup>2</sup> Ibid.

<sup>3</sup> Ibid.

<sup>4</sup> Ibid.

Next, add sugar, mustard flour, salt, paprika and pepper, and beat until creamed. This will require from 2 to 5 minutes, depending on the per cent of egg white that adheres to the yolks. The more whites, the longer the time required and also the less desirable.

Now start the oil slowly at first, and then add at the rate of 1 gal. per minute, until 9 gals. have been added.

The beater is then run on slow speed while 1 qt. of vinegar and 1 qt. of water are added and thoroughly mixed in.

Now run beater on high speed and slowly add remainder of oil at the rate of 1 gal. every 2 minutes. Shift beater to slow speed, and then add the remainder of the vinegar and water.

Shut off beater. Scrape down sides of bowl. At slow speed, mix entire batch for 2 minutes longer. Long beating of the entire mix tends to make the mayonnaise thinner.<sup>5</sup>

### FORMULA No. 6. (Modified)

Salad Oil	20	lb.	Mustard	21/2	oz.
Egg Yolk	2	lb.	Concentrated Lemon Juice	3	oz.
100-Grade Pectin (Citrus)	11/2	oz.	Water	14	oz.
Sugar	43/4	oz.	Vinegar <sup>6</sup>	20	0 <b>z</b> .
Salt	43/4	OZ.	_		

#### MAYONNAISE

#### FORMULA No. 7.

Egg Yolks	14	1b.	Salt	16.
Vinegar	10	1b.	Sugar	1/2
Cotton Seed Oil (Prime			Mustard	3/4
Summer Yellow)	70	lb.	Pepper 7	1/4

Mix thoroughly in mixing bowls and run through a colloid mill with a clearance of .005".

<sup>&</sup>lt;sup>5</sup> Bennett, Chem. Formulary, Vol. 5, p. 147 (1941).

<sup>6</sup> Ibid.

<sup>&</sup>lt;sup>7</sup> Ibid., Vol. 4, p. 133 (1939).

#### SALAD DRESSINGS

#### FORMULA NO. 1.

Cornstarch	5	Salt	5
Water	90	Sugar	10
Cottonseed Oil	51	Vinegar (White)	42
French Mustard	25	,	72

Mix the cornstarch with a little cold water and add to the remaining hot water. Stir and boil until it stiffens, set aside to cool. Put the mustard into a bowl and gradually add the oil, stirring all the time. Next add sugar, salt, vinegar and then the cooled-down cornstarch 1

#### FORMULA No. 2.

Tapioca Starch	7	Salt	2
Water	30	Cane Sugar	4
Corn or Cottonseed Oil	40	Vinegar (White 5%)2	17

#### FORMULA No. 3.

Whole Eggs Mazola (Corn Oil) No. 2 Buffalo Starch Water Vinegar	9 12 pt. 2.4 lb. 2 gal. 7 pt.	Cerelose (Corn Sugar) Salt Mustard Paprika <sup>8</sup>	2.4 lb. 9.6 oz. 9.6 oz. 0.96 oz.
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### FORMULA No. 4.

Whole Eggs Mazola No. 2 Buffalo Starch Water Vinegar	11 16 1.8 11 7	pt. lb. pt. pt.	Cerelose Salt Mustard Red Pepper 4	2,4 9,6 9,6 0,96	1b. 1b.
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### WHITE OR CREAM SAUCE

Butter Gum Karaya Heavy Cream	3 tbsp. 2 tbsp. ½ cúp	Water Salt and Pepper	½ cup To taste
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Handbook of Food Mfg., Fiene and Blumenthal.
 Handbook of Food Mfg., Fiene and Blumenthal.

<sup>&</sup>lt;sup>8</sup> Crowley, Money Making Formulas. 4 Crowley, Money Making Formulas.

Heat butter and mix with water and cream. When heated gradually sift in the gum karaya, beating or stirring until the gum is dissolved and season. Serve hot. If thin sauce is desired, use only 1 tablespoonful of gum.<sup>5</sup>

<sup>&</sup>lt;sup>5</sup> Fiene and Blumenthal, Handbook of Food Manufacture.

### CHAPTER IX

### GASOLINE EMULSIONS

FORMULA No. 1.

Triethanolamine Water Oleic Acid	1½ 1 1½	Butanol Gasoline	5 <b>4</b> 5
Dissolve triethanol other ingredients slov			
	Formula	No. 2.	
Triethanolamine Water Oleic Acid	1 1 1	Butanol Gasoline	5 45
	FORMULA	No. 3.	
Triethanolamine Water Oleic Acid	1/2 1 1/2	Butanol Gasoline	5 45
	FORMULA	No. 4.	
Triethanolamine Oleic Acid Water	1 1 1	Butanol Gasoline	5 45
	FORMUL	A No. 5.	
Triethanolamine Water Oleic Acid	1 1 1	Butanol Alcohol	2 4

<sup>\*</sup> The stability of the above emulsions is improved considerably if they are passed through a colloid mill.

	Gasoline Emulsions	<b>30</b> 9
	FORMULA No. 6.	
Triethanolamine Water Oleic Acid	1 Butanol 1 Alcohol 1	4 2
	Formula No. 7.	
Triethanolamine Water Oleic Acid	1 Butanol 1 Kerosene 1 Gasoline	5 20 25
	Formula No. 8.	
Triethanolamine Water Oleic Acid	1 Alcohol 1 Gasoline 1	3 45
	FORMULA No. 9.	
Triethanolamine Water Stearic Acid	1 Alcohol 1 Gasoline 5	3 45
	FORMULA No. 10.	
Triethanolamine Water Stearic Acid	1 Alcohol 1 Gasoline 3	3 45
	FORMULA No. 11.	
Triethanolamine Water Stearic Acid	1 Alcohol 1 Gasoline 2	3 45
	FORMULA No. 12.	
Triethanolamine Water Stearic Acid	1 Alcohol 1 Gasoline 3	2 45
	FORMULA No. 13.	
Triethanolamine Water Stearic Acid	1/2 Alcohol 1 Gasoline 3	3 45
	FORMULA No. 14.	
Triethanolamine Water Stearic Acid	1/4 Alcohol 1/2 Gasoline	3 45

310	Practical .	Emulsions				
	Formula	No. 15.				
Triethanolamine Water Stearic Acid	1/4 1/2 2	Alcohol Gasoline	2 45			
	Formula	No. 16.				
	1% V	Vater				
Triethanolamine Water Stearic Acid	175 350 1400	Alcohol Gasoline	1400 31500			
	Formula	No. 17.				
	34% V	Water				
Triethanolamine Water Stearic Acid	175 260 1400	Alcohol Gasoline	1400 31500			
	Formula	No. 18.				
	½% V	Water				
Triethanolamine Water Stearic Acid	175 175 1400	Alcohol Gasoline	1400 31500			
*	Formula	No. 19.				
	14%	Water				
Triethanolamine Water Steazic Acid	175 85 1400	Alcohol Gasoline	1400 31500			
	Formula	No. 20.				
10% Water						
Trihydroxyethylamine Laurate Gasoline	3500 31500	Butanol Water Triethanolamine	5600 3500 1 <b>7</b> 50			
	Formula	No. 21.				
5% Water						
Trihydroxyethylamine Laurate Gasoline	2100 31500	Butanol Water Triethanolamine	3500 1 <b>7</b> 50 1050			

	Gasoline E	Emulsions	311		
	FORMULA	No. 22.			
	1% W	ater			
Trihydroxyethylamine Linoleate Gasoline	2100 31500	Butanol Water Triethanolamine	3500 700 1050		
,	Formula	No. 23.			
Gasoline Alcohol	70.0 9.0	Water Bentonite <sup>23</sup>	20.3 0.7		
	Formula	No. 24.			
Gasoline Water	80.0 19.3	Bentonite <sup>24</sup>	0.7		
ANTI-KNOCK FUEL					
Triethanolamine Oleic Acid Butyl Cellusolve	18 58 20	Sec. Hexyl Alcohol Water Gasoline <sup>25</sup>	25 75 750		
5	SOLIDIFIED	GASOLINE			

#### FORMULA NO. 1.

Coconut Oil	50 g.	Water	120 cc.
Caustic Soda Solution		Benzol	60 cc.
(10%)	46 cc.	Gasoline	5 gal.

Mix the coconut oil, caustic solution, alcohol, and 20 cubic centimeters of the water, and heat over a Bunsen burner until completely saponified. Then add the remaining 100 cubic centimeters of the water and stir out. Add to this the benzol and stir until completely emulsified. Then add the gasoline in small portions while stirring vigorously with a mechanical stirrer. The resulting jelly-like mass forms a very stable emulsion.26

<sup>1-22</sup> Inclusive: Bennett, Chem. Formulary, Vol. 3, p. 107 (1936).

<sup>28</sup> P. C.

<sup>&</sup>lt;sup>25</sup> Bennett, Chem. Formulary, Vol. 5, p. 603 (1941). <sup>26</sup> Ibid., Vol. 2, p. 237 (1935).

#### FORMULA No. 2.

Stearic Acid	35	Alcohol	500
Caustic Soda (30%)	7	Gasoline <sup>27</sup>	4500

### FORMULA No. 3.

Gasoline	0.5 gal.	Water	1.0 pt. 5
White Soap (Fine		Household Ammonia	5 oz.
Shaved)	12 oz.		

Heat the water, add soap, mix and when cool add the ammonia. Then work in slowly the gasoline to form semi-solid mass.<sup>28</sup>

#### FORMULA No. 4.

#### SOUND ABSORBING COMPOSITION

Exfoliated mica is used with the following binder:

Casein	25 lb.	Caustic Soda (15%)	10 lb.
Water	<sup>3</sup> 2 lb.	Alum	2½ 1b.

After the casein has swelled and mixed until smooth, run in slowly with good mixing

Water 57½ lb. Gasoline 57½ lb.

Put into forms and allow to dry.

<sup>&</sup>lt;sup>27</sup> Klinkenstein, U. S. 1,848,568 (1932).

<sup>&</sup>lt;sup>28</sup> Bennett, Chem. Formulary, Vol. 4, p. 15 (1939).

#### CHAPTER X

### LACOUER EMULSIONS

Combinations of a lacquer with an emulsifying mixture results in some degree of emulsification and can be realized by any customary method. Most useful concentration ratio is  $2\frac{1}{2}$  lacquer to 1 of water phase.

Stability generally varies with particle size—all other variables being constant.

Poorest emulsions are made by rapid stirring with a high speed mixer. They are greatly improved by a stator-rotor type sheering action, of a colloid mill. The most stable emulsions are usually produced by high pressure extrusion, i.e., in an homogenizer. For easily dispersed systems, an homogenizer alone is sufficient. More difficulty dispersable systems must be predispersed by other types of equipment (colloid mill) before passing through homogenizer.

An emulsion with many large aglomerate particles can in general be expected to have a short life. The dispersed droplets should be small and uniform.

Almost any good emulsifying agent, and this includes soap, will form some type of an emulsion with a lacquer. Useful stabilities and this means three months or better, can only be realized by a careful selection of the emulsifier. One of the best found contains 0.5% Dupanol, 1.0% sulfonated

castor oil either alone or mixed with other emulsifiers or lacquer solvents.

Lacquers rich in resins are more easily dispersed than those that are nitro-cellulose rich, and as a rule have a longer life (Table A No. 2 and 5).

Lacquer emulsions have high flash points and are usually broken by freezing. Lacquers of very high viscosity are difficult to disperse.

#### NITROCELLULOSE EMULSIONS

Table A
LACQUER BASE FORMULAE (CLEAR)

FORMULA NO.	1	2	3	4	5
Nitro Cellulose (Dry Weight)	30	25	30	30	40.0
Castor Oil					
Triethyl Citrate		20			5.0
Tricresyl Phosphate			12		
Dibutyl Phthalate		• •		13	
Resin (Alkyd)		10	15	15	
Butyl Acetate		25	25	22	30.0
Xylene		5			7.5
Butanol		15	18	20	17.5

#### FORMULA No. 6.

Pigmented Nitrocellulose		Xylene	14.4
(Dry Weight)	20	Octyl Acetate	10
Resin (Alkyd)	8	Butyl Acetate	25
Castor Oil (Blown)	6	Alcohol (Present in nitro	
Diamyl Phthalate	6	cellulose)	8.6
Carbon Black	2	•	

### Table A

Any of these formulae can be emulsified without much difficulty using any of the emulsifiers of Table B. The difference lies in the properties of the dried films.

They are all formulated electrolyte free—small amounts can be included and dispersed. The presence of gelatin or

casein in the water phase will help disperse a certain concentration of electrolytes.

### LACQUER EMULSIFIERS

#### TABLE B

	FORMULA	No. 1.	
Duponol Sulfonated Castor Oil	0.5% 1.0	Water	98.5
	FORMULA	No. 2.	
Duponol Sulfonated Castor Oil	0.5% 1.0	Acetone Water	10.0 88.5
	FORMULA	No. 3.	
Duponol Sulfonated Castor Oil	0.5% 1.0	Casein Water	0.5 98.0
	FORMULA	No. 4.	
Duponol Sulfonated Castor Oil	0.5% 1.0	Gelatin Water	2.0 96.5
	FORMULA	A No. 5.	
Duponol Sulfonated Castor Oil Casein	0.5% 1.0 0.5	Acetone Water	10.0 88.0
	FORMULA	A No. 6.	
Sodium Oleate	6%	Water	94
	Formula	A No. 7.	
Duponol Sulfonated Castor Oil	0.5% 1.0	Methyl Cellulose Water	0.3 98.2

Illustrates many of the various possible combinations of emulsifiers.

1. The emulsion stability varies with the composition of the lacquer phase. The presence of resins in lacquer usually aids the dispersion, both as to the ease of forming the emulsion and the uniformity of droplet size.

- 2. The presence of acetone gives (1) increased emulsifying power for n/c rich lacquers, (2) depresses freezing temperature of the emulsion, (3) acetone replaced by a water miscible high boiling solvent decreases the tendency of film blush.
- 3, 4, 5, 8 are all of the same general type, i.e., they contain an auxiliary emulsifier or stabilizer. This additional material helps produce emulsions from lacquers of very high viscosity and those that are relatively rich in n/c.
- 6. While the stability of the emulsion using this emulsifying mixture may be satisfactory, too high a concentration of the emulsifiers that are incompatible with the lacquer results in a decrease of gloss upon the surface of the dried films.
- 7. Will produce a W/O emulsion. If the concentration is decreased to get a O/W emulsion a poor emulsion results (only partially emulsified).

Lacquer emulsions with casein emulsions are O/W; gelatin emulsions are W/O, 0.03% is the upper limit of methyl cellulose that can be included without producing "flatting" of the film. Methyl cellulose is an exceptionally good emulsifying aid.<sup>1</sup>

#### FORMULA No. 7a.

LACQUER PHASE		WATER PHASE	
Nitrocellulose, (15-20 seconds		Water	86.5
viscosity, butanol		Acetone	10.0
dehydrated)	19.3	Duponol ME	0.5
Butanol	4.0	Sulfonated Castor Oil	1.0
Petrex #5	8.0	Casein	1.0
Dibutylphthalate	10.8	Borax (5% Solution)7	1.0
Titanox A	13.5	,	
Butyl Acetate	12.3		
Pent Acetate	12.3		
Toluol	19.8		

<sup>7</sup> Hercules Powder Co., Wilmington, Del.

### FORMULA No. 8.

	WATER PHASE	
	Duponol ME (Dry)	0.5
	Sulfonated Castor Oil	1.0
28.6	Methyl Cellulose	0.3
80	Acetone	10.0
2.0	Water 8	88.2
2.0		
11.5		
11.5		
11.5		
24.9		
	80 2.0 2.0 11.5 11.5	Duponol ME (Dry) Sulfonated Castor Oil 28.6 Methyl Cellulose 80 Acetone 2.0 Water 8 2.0 11.5 11.5

#### FORMULA No. 9.

LACQUER PHASE		Water Phase	
Nitrocellulose, 15-20 seconds		Duponol, ME	0.5
viscosity, butanol,		Sulfonated Castor Oil	1.0
dehydrated	21.4	Casein	1.0
Titanium Dioxide	4.0	Borax (5% solution)	1.0
Chrome Yellow	4.0	Acetone	10.0
Dibutylphthalate	8.0	Water 9	86.5
Petrex 5	10.0		
Sec. Hexyl Acetate	16.4		
Pent Acetate	3.6		
Hexone	7.3		
Xylol	11.8		
Toluol	11.0		
Butanol	2.5		

### FORMULA No. 10.

LACQUER PHASE		WATER PHASE	
RS ½" N.C. (Dry wt.)	24.0	Duponol ME (dry)	0.5
Glyptal 2471 (Solid)	12.0	Sulfonated Castor Oil	1.0
Tricresyl Phosphate	14.0	Acetone	10.0
Butanol carried by N.C.	10.3	Water 10	88.5
Toluol	14.7		
Butvl Acetate	12.5		
Hexone	12.5		

### FORMULA No. 11.

The solids content of the lacquer base can be increased to 60 per cent in special formulations by using low-viscosity

<sup>8</sup> Ibid.

<sup>9</sup> Ibid.

<sup>10</sup> Ibid.

type nitrocellulose, a solvent plasticizer, and an exceptionally good solvent mixture. This allows a 1:1 ration of solvent to water. The formulation below is a typical example of such a composition.

LACQUER PHASE RS ½" N.C. (Dry wt.) Glyptal 2471 (Solid) Dibutyl Phthalate Butanol carried by N.C. Toluol Secondary Hexyl Acet. Hexone	30.0 13.0 17.0 12.9 13.0 7.0 7.1	WATER PHASE Duponol ME (Dry) Sulfonated Castor Oil Acetone Water 11	0.5 1.0 10.0 88.5
F	ORM ULA	No. 12.	
LACQUER PHASE RS 5-6" N.C. (Dry Wt.) Glyptal 2471 (Solid) Tricresyl Phosphate Butanol carried by N.C. Toluol Xylol Hexyl Acetate Hexone	21 8 12 9 12 12 12 12	WATER PHASE Duponol ME (dry) Sulfonated Castor Oil Acetone Water 12	0.5 1 10 88
F	ORMULA	No. 13.	
LACQUER PHASE R.S. 15-20" N.C. (Dry wt.) Ester Gum Dibutyl Phthalate Butanol carried by N.C. Toluol Butyl Acetate Hexone	18.0 9.0 8.0 7.8 27.2 15.0 15.0	WATER PHASE Duponol ME (Dry) Sulfonated Castor Oil Acetone Water <sup>23</sup>	0.5 1.0 10.0 88.5
BLACK PIGMEN	TED I	LACQUER EMULSION	
LACQUER PHASE R.S. 1/2" sec. (Dry weight) Dammar Gum (Dewaxed) (Dry Weight) Dibutyl Phthalate Tricresyl Phosphate	20.0 6.0 3.0 2.6	WATER PHASE Water Duponol M.E. (dry) Sulphonated Castor Oil	98.50 0.25 1.25

<sup>11</sup> Ibid.

<sup>₽</sup> Ibid.

<sup>18</sup> Ibid.

# Lacquer Emulsions

LACQUER PHASE	
Blown Castor Oil	5.4
Carbon Black	2.0
Butanol carried by Nitro-	
cellulose	8.6
Butanol	5.0
Denatured Ethyl Alcohol	
(in Dammar Solution)	4.0
Hi-Flash Solvent Naphtha	13.8
Toluol (in Dammar solution)	2.0
Secondary Hexyl Acetate	13.8
Octyl Acetate	13.8
-	

Ratio of lacquer phase to water phase recommended: From 2.5 to 1, to 3 to 1 parts by weight.<sup>14</sup>

# GRAY PIGMENTED LACQUER EMULSION

LACOUER PHASE		WATER PHASE	
R.S. ½ sec. (Dry weight)	18.0	Water	98.20
Dammar Gum (Dewaxed)		Duponol M.E. (dry)	0.25
(Dry weight)	5.4	Sulphonated Castor Oil	1.25
Tricresyl Phosphate	3.0	Methyl Cellulose	0.30
Blown Castor Oil	6.0		
Gray Pigment *	12.0		
Butanol carried by Nitro-			
cellulose	7.8		
Butanol	1.9		
Denatured Ethyl Alcohol			
(in Dammar Solution)	4.0		
Hi-Flash Solvent Naphtha	14.0		
Toluol (in Dammar solution)	2.0		
Secondary Hexyl Acetate	13.0		
Octyl Acetate	12.9		

Ratio of lacquer phase to water phase recommended: From 2 to 1, to 2.5 to 1 parts by weight.<sup>15</sup>

# \*COMPOSITION OF GRAY PIGMENT

Zinc Oxide Chrome Yellow Carbon Black		Prussian Blue Iron Oxide	0.7 oz. 1.0 oz.
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<sup>14</sup> Bennett, Chem. Formulary, Vol. 4, p. 97-98 (1939).

<sup>15</sup> Ibid.

# CHAPTER XI

# LEATHER TREATMENT EMULSIONS

	***************************************		
	FAT LI	QUORS	
	Formula	No. 1.	
Paraffin Oil Sulphonated Cod Oil	60 lb. 40 lb.	Borax Water (at 140°F.) <sup>1</sup>	2 lb. 20 gal.
	FORMUL	A No. 2.	
Fig Soap Water Waterless Moellon	30 gal.	Sulphonated Cod Oil Paraffin Oil Water, <sup>2</sup> To	20 lb. 30 lb. 50 gal.
	FORMUL	A No. 3.	
Soap Neatsfoot Oil		Soda Water <sup>3</sup>	97 <sup>1</sup> ⁄ <sub>4</sub>
	FORMUL	A No. 4.	
Sulphonated Cod Oil Stock Solution made as be Cod Oil Degras	elow 2.1b.	Soap Bicarbonate of Soda Water 4	6 1b. 3 1b. 5500 1b.
	FORMUL	No. 5.	•
Sulphonated Neatsfoot Oil	20-40 lb.	Spindle Oil Neatsfoot Oil	10-20 lb. 100-80 lb.

<sup>&</sup>lt;sup>1</sup> Bennett, Chem. Formulary, Vol. 2, p. 288 (1935).

<sup>&</sup>lt;sup>2</sup> Ibid., Vol. 2, p. 289 (1935). <sup>3</sup> Ibid., Vol. 4, p. 159 (1939).

<sup>4</sup> Ibid.

Leather	Treatment	Emulsions

321

	No. 6	No. 7	No. 8
Sulfonated Neatsfoot Oil	30	20	25 lb.
Neatsfoot Oil	40	40	30 lb.
Mineral Oil	20	20	20 lb.
Soap	10	20	25 lb.

5% emulsions of the above are used at pH of 6.5-8.0.5

#### FORMULA No. 9.

Water (150°F.)	1 gal.	Light Specific Gravity	
Sulphonated Cod Oil	2 lb.	Mineral Oil	2 lb.
Raw Cod Oil	1 lb.	Tallow Soap, Dissolved in	
		1 Gal. Water 6	1 lb.

#### FORMULA No. 10.

Cold Test (20°)	Neatsfoot	Water	25 gal.
Oil		Sulphonated Castor Oil	
Paraffin Oil (28°)	25 gal.	(50%)?	25 lb.

Mix water first with the sulphonated castor oil. Then mix all ingredients at 30°C.

#### LEATHER DRESSINGS

#### FORMULA No. 1.

Tallow	70.0	Beeswax	9.0
Petroleum Jelly	3.5	Rosin	2.0
Diglycol Stearate	13.0	Water 8	2.0

#### FORMULA No. 2.

Water Glass (40-45° Bé.) Linseed Oil

# Mix until emulsified. Apply with brush.9

Water	15 gal.	Methylated Spirits	1 qt.
Castor Oil	5 qt.	Benzol	1 qt.
Casein Solution	4 qt.	Lactic Acid 10	½ pt.

<sup>&</sup>lt;sup>5</sup> Ibid., p. 190.

<sup>6</sup> Ibid., p. 157. 7 Ibid., Vol. 3, p. 200 (1936).

<sup>8</sup> P. C.

<sup>&</sup>lt;sup>9</sup> Ibid., Vc<sup>1</sup> <sup>1</sup> p. 424 (1933).

<sup>10</sup> Ibid., p.

#### LEATHER FINISHES AND POLISHES

#### FORMULA No. 1.

Beeswax	5	Ammonia	1
Grey Fatty Carnauba Wax	1	Water	100
Olive Oil Bar Soap	5	Glycerin	1

The soap is dissolved in about 50 hot water and the ammonia added. The wax mixture is melted separately and added with stirring to the soap solution. The temperature of the soap solution must be well above the setting point of the wax, preferably about 90°C. When the emulsion is satisfactorily made the remainder of the water and the glycerin can be added and the mixture cooled down. A mixture of beeswax and carnauba is used here because the beeswax alone is too soft and sticky, while the carnauba alone would present difficulties in emulsification. Such a mixture is suitable for application to shoe calf and gives a soft brush-polished finish.<sup>1</sup>

#### FORMULA No. 2.

Castile Soap	15 lb.	Candeililla Wax	5 lb.
Carnauba Wax (No. 1			
Yellow)	5 lb.		

Dissolve the soap in 8 gallons of water by boiling for one hour, adding water as it is lost by evaporation, then add melted wax and continue boiling for two more hours, then add water enough to make a total of 10 gallons. Strain the mass through 4 layers of cheese cloth.<sup>2</sup>

#### FORMULA No. 3.

Gray Fatty Carnauba W	ax 20 lb.	Water	16 gal.
Turpentine	10 lb.	Turkey Red Oil (50%)	20 gal.
Olive Oil Soap	10 lb.	- (,-,	

The carnauba wax and turpentine are melted in a steam jacketed mixing pan. The soap, previously dissolved in the

<sup>&</sup>lt;sup>1, 3, 4</sup> Davidsohn-Davidsohn, Shoe Finishes & Polishing Waxes. <sup>2</sup> Bennett, Chem. Formulary, Vol. 4, p. 178 (1939).

hot water, is added with constant stirring and the mixture boiled for ten minutes, when the steam can be turned off and the Turkey red oil added. The hot liquid is run through a water-cooled colloid mill which cools and emulsifies it at the same time. A modification of the above mixing makes use of glue as a stabilizing colloid for the soap-wax emulsion and may be prepared in a similar manner.<sup>3</sup>

#### FORMULA No. 4.

Grey Fatty Carnauba Wax	20 lb.	Water	16 gal.
Turpentine	1.0 lb.	Rabbit Skin Glue	40 lb.
Olive Oil Soap	10 lb.	Water 4	16 gal.

#### SHOEMAKERS' WAX BURNISHING POLISH

Carnauba Wax	10.0	Iron Chloride or Acetate	3.0
Turpentine '	10.0	Acetic Acid (10%)	2.0
Duponol WA Paste	10.0	Water	65.0

Melt the wax and turpentine together and pour into a solution of the DUPONOL in 45 parts of the water at 95–100°C. Stir vigorously until a smooth emulsion is formed and then stir gently until the emulsion has cooled to approximately 35°C. At this point add a solution containing the iron salt and acetic acid, dissolved in the remaining 20 parts of water. Continue stirring slowly. The viscosity of the emulsion will be at first greatly increased after which it will return to its initial value; the resulting emulsion is a smooth, fluid paste <sup>5</sup>

#### FORMULA No. 5.

Shellac Wax	3	Carbon Tetrachloride	20
Montan Wax	3	Water	20
Carnauba Wax	3	Soda	3
Paraffin Wax	2	Castor Oil	1
Japan Wax	1	Naphtha	5
Varnish, Acetone	1	Turpentine Substitute	40
Lacquer, Nitrocellulose	1	Bornyl Acetate	1
Lacquer, Acetate	1	Shellac <sup>6</sup>	1

<sup>&</sup>lt;sup>5</sup> E. I. du Pont de Nemours Co., Wilmington, Del.

<sup>6</sup> Veith, Brit. 425,323 (1935).

#### EDGE FILLER FOR SHOE FACTORY USE

Soap	15 lb.	Gelatine	11½ lb.
Yellow Dextrin	5½ 1b.	Formaldehyde	1 qt.
Neatsfoot Oil	1½ qt.	Water	1 qt.
Oil of Mirbane	1 pt.		

This is made up with sufficient water to make 60 gal. solution.

<sup>&</sup>lt;sup>7</sup> Bennett, Chem. Formulary, Vol. 3, p. 205 (1936).

# CHAPTER XII

# LUBRICANT EMULSIONS

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#### FORMULA No. 1.

#### White

a. Stearic Acid	10	b. Lime Hydrate	
Paraffin Oil, white	30	Water	2
		c. Paraffin Oil, White	56

Melt a, saponify hot with b; add c in small portions. Keep stirring until cold.<sup>1</sup>

#### FORMULA No. 2.

#### Yellow

a. Train Oil Fatty Acid	16	b. Lime Hydrate	
Mineral Oil, Pale	30	Water	2
,		c. Mineral Oil. Pale	48

Prepare same as above.1

#### FORMULA No. 3.

Green Petrolatum	250 1Ь.	Fat	55 lb.
Paraffin Pale Oil (28°	Bé.) 92 gal.	Water	3 gal.
Lime	9 lb.		

<sup>&</sup>lt;sup>1</sup> Bennettt, Chem. Formulary, Vol. 4, p. 192 (1939). <sup>1a</sup> Ibid.

Melt the petrolatum in the mineral oil. Mix well, then proceed as for ordinary calcium soap grease.<sup>2</sup>

Train Oil Fatty Acid	12	Spindle Oil	82
Lime, Hydrated	2	Water 3	2
Zinc Oxide	2		

#### "COLD ROLLER GREASE BRIQUETS"

#### FORMULA No. 1.

Crude Woolfat	15	Cylinder Oil	
Stearin from Woolfat	5	(7 E. at 100°C.)	78
		Caustic Soda (40° Bé.) <sup>3a</sup>	6

#### FORMULA No. 2.

Tallow	20	Cylinder Oil	
Caustic Soda (40° Bé.)	8.5	(7 E. at 100°C.)4	77

#### HORSE-SHOE GREASE

#### FORMULA No. 1.

Woolfat, Neutral	5.2	Paraffin Wax	0.3
Spindle Oil	2.0	Water 1	About 2.4
Soft Tar	0.5		

#### MIXED BASE GREASE

Lime	17 lb.	Aluminum	Oleate (Pulp	
Fat	113 lb.	Stock)		50 lb.
Pale Oil (100 Viscosity)	112 gal.	Water		6 gal.

Place the fat in a steam jacketed kettle equipped with paddles for stirring, add a small portion of the mineral oil, mix the lime with sufficient water to form a thin paste and add this to the material in the kettle. Turn on the steam and start the paddles. When the soap has cooked for 5 hours it should be tested to determine if saponification is completed, if so the steam is turned off and half of the balance of the

<sup>&</sup>lt;sup>2</sup> P. C.

<sup>3</sup> Ibid., Vol. 3, p. 213 (1936).

<sup>&</sup>lt;sup>3a</sup> Ibid., Vol. 4, p. 195 (1939).

<sup>4</sup> Ibid.

<sup>&</sup>lt;sup>1</sup> Bennett, Chem. Formulary, Vol. 4, p. 203 (1939).

mineral oil is run in slowly. The rest of the mineral oil is run into a separate kettle and the aluminum oleate melted in it and this mixture is pumped into the first kettle while still warm. Stirring should be continued until a smooth uniform grease is produced.<sup>2</sup>

#### SOAP BASE LUBRICATING EMULSION

Cottonseed Oil	3 kg.	Caustic Soda	132 g.
Mineral Oil	1-2 kg.		_

Heat to 180°C. until foaming stops. Add 13 kg. mineral oil in successive portions at intervals with stirring bringing up to 190–210°C. Pour into wooden tubs and cool to 70°C. Add 9 kg. water with stirring.<sup>8</sup>

#### VISCOUS EMULSION LUBRICANT

Heavy Mineral Oil	10	Water	40
Diglycol Stearate	10		

Heat together to 60°C. and stir until cold. A heavy cream results. The color is dependent on the color of the oil used.<sup>4</sup>

#### WATER IN OIL EMULSION LUBRICANT

50 grams of a heavy paraffin oil is mixed with 1 g. of n-lauryl-l-sulphuric acid and stirred with 0.25 g. of lead acetate dissolved in 48.75 g. of water. The resulting emulsion is a heavy grease and has good lubricating properties and may readily be extended with oil.<sup>5</sup>

#### GRAPHITE LUBRICANT

Degras (Free from Fatty		Turpentine	8.7
Acids)	20	Ammonia (28%)	4.4
Kerosene	16	Graphite Powder 6	30
Water	75		

<sup>&</sup>lt;sup>2</sup> Ibid., Vol. 3, p. 209 (1936).

<sup>&</sup>lt;sup>8</sup> Ibid., p. 111.

<sup>4</sup> Ibid., Vol. 2, p. 302 (1935).

<sup>&</sup>lt;sup>5</sup> Ibid., Vol. 4, p. 210 (1939).

<sup>6</sup> Ibid., Vol. 3, p. 213 (1936).

# CHAPTER XIII

# MEDICINAL EMULSIONS

***************************************			·······		
BUR	N OIN	TMENTS			
F	ORMULA	No. 1.			
Lanolin Olive Oil Spermaceti	30 25 10	Cycloform Water <sup>1</sup>	10 25		
F	ORMULA	No. 2.			
Glyceryl Monomyristate Butyl Myristate Olive Oil Alcohol	10.0	Cycloform Betanaphthol Water <sup>2</sup>	10.0 2.5 50.0		
COD LIV	VER O	IL EMULSION			
F	FORMULA	No. 1.			
Water	26 oz. 44 oz. 50 g. 5 oz.	Calcium Hypophosphite Sodium Hypophosphite Saccharine <sup>1a</sup>	½ oz. ½ oz. .001 oz.		
F	Formula No. 2.				
a. Cod Liver Oil Gum Arabic Gum Tragacanth b. Gelatin Water	420 15 7½ 1 299	Water S Water	12 (90 (60 86		
<sup>1</sup> Colloid Chem. Labs., Jers	ey City,	N. J.			

<sup>18</sup> Bennett, Chem. Formulary, Vol. 1, p. 379 (1933).

Make up a by grinding, and b by heating. Add b to a at once and stir until cooled.

To the cooled emulsion add the cooled solution c slowly, with good agitation.<sup>2</sup>

#### FORMULA No. 3.

h	Gum Arabic Gum Tragacanth Glycerin Cod Liver Oil, Crude	100 100–120 1200 3700	Calcium Hypophosphite Sodium Hypophosphite Water	50 50 4000
b.	Cod Liver Oil, Crude	3700		

Grind a until smooth, add b in small portions, homogenizing every time. To this add c in an emulsifying machine.

#### FORMULA No. 4.

Gum Arabic .	12	Cod Liver Oil	300
Gum Tragacanth	16	Spice Oil Mixture	10
Glycerin (28° Bé.)	140	Calcium Hypophosphite	8
Distilled Water	430	Sodium Hypophosphite	12
Moldex or Other Good		Distilled Water 4	71
Preservative	1		

#### FORMULA No. 5.

a. Caragheen Moss	15.0	b. Starch Syrup	8.8
Water	30.0	Water	9.0
Cod Liver Oil	50.0	Spice Decoction 5	0.1
Preservative	0.1	-	

#### FORMULA No. 6.

Carragheen Moss Distilled Water Moldex or Other Good	300	Cod Liver Oil Syrup, White Distilled Water	500 86 91
Preservative	1	Spice Oil Mixture 6	10

#### FORMULA No. 7.

Gum Arabic	12	Water, Distilled	340
Gum Tragacanth	12	Sodium Salicylate 7	5
Glycerin (28° Bé.)	130		

<sup>&</sup>lt;sup>2</sup> Ibid., Vol. 4, p. 114 (1939).

<sup>&</sup>lt;sup>3</sup> Bennett, Chem. Formulary, Vol. 3, p. 126 (1936).

<sup>&</sup>lt;sup>4</sup> Ibid., p. 125.

<sup>&</sup>lt;sup>5</sup> Ibid., Vol. 4, p. 115.

<sup>&</sup>lt;sup>6</sup> Bennett, Chem. Formulary, Vol. 3, p. 125 (1936).

<sup>7</sup> Ibid.

#### FORMULA No. 8.

Cod Liver Oil		Benzaldehyde	1 cc.
Water	88 oz.	Coriander Oil	1 cc.
Glycerine	10 oz.	Cardamom Oil	2 cc,
Tragacanth	100 g.	Tincture Vanilla !	5 cc.
Sassafras Oil	5 cc.		

#### FORMULA No. 9.

Calcium Hypophosphite Sodium Hypophosphite Sodium Chloride Gum Acacia Elixir of Gluside	Almond Oil Glycerin Cod-Liver Oil Distilled Water, <sup>9</sup> To make	15 min. 2 fl. oz. 8 fl. oz. 16 fl. oz.

#### FORMULA No. 10.

Calcium Hypophosphite Sodium Hypophosphite Gum Acacia Cod Liver Oil	32 gr. 32 gr. 1 oz. 4 fl. oz.	Elixir of Gluside Cassia Oil Distilled Water, <sup>10</sup> To make	2	min. min. fl. oz.
	Formula	No. 11.		

#### FORMULA No. 11. 4 fl. oz. Gluside

1 gr.

Gum Acacia	480 gr.	Distilled Water,11 To	_
Cassia Oil	4 min.	make	8 fl. oz.

Mix the cod-liver oil and oil of cassia with the gluside and gum acacia in a dry mortar; add two fluid ounces of distilled water, and triturate until an emulsion is formed. Add gradually, with constant trituration, sufficient distilled water to produce eight fluid ounces of mixture.

#### FORMULA No. 12.

Calcium Hypophosphite		Clove Oil	6 min.
Sodium Hypophosphite	160 gr.	Lemon Oil	20 min.
Gum Acacia	2 oz.	Chloroform	15 min.
Tragacanth	1/2 oz.	Alcohol	1 fl. oz.
Cod-Liver Oil	40 fl. oz.	Distilled Water 12 To	
Elixir of Gluside	4 fl. oz.	make	80 fl. oz.
Almond Oil	6 min.		

<sup>8</sup> Ibid., Vol. 1, p. 379 (1933).

Cod Liver Oil

<sup>9</sup> P. C.

<sup>10</sup> P. C.

<sup>11</sup> P. C.

<sup>12</sup> P. C.

#### FORMULA No. 13.

Tragacanth Alcohol Almond Oil Cod Liver Oil	3	gr. fl. oz. min. fl. oz.	Lemon Oil Syrup Distilled Water <sup>13</sup>	40 min. 1 fl. oz. 12 fl. oz.
	F	ORMULA	No. 14.	
Cod Liver Oil Pectin		80.0 0.5	Milk Sugar Water 14	20.0 20.0

#### FORMULA No. 15.

75 grams gum arabic powder is dissolved in 900 cubic centimeters water. This solution is placed in a tank fitted with mechanical stirrer, and 600 cubic centimeters cod liver oil are added. Stirring is continued for about a quarter of an hour, when a good primary emulsion is made. Further ingredients, such as oil of bitter almonds, hypophosphites, glycerophosphates, etc., are added, and the primary emulsion is fed to a De Laval homogenizer. Homogenization is carried out at a pressure of 3,500 pounds per square inch, when an absolutely stable and even emulsion is obtained of a suitable viscosity for pouring from a bottle.<sup>15</sup>

#### FROST BITE PREVENTION CREAMS

#### FORMULA No. 1. Lanolin 10.0 Water 25.0 Cocoa Butter 15.0 Borax 1.5 Benzoic Acid Olive Oil 25.0 0.5 White Beeswax 22.0 Perfume 1 1.0 FORMULA No. 2. Almond Oil 425.0 Spermaceti 62.0 Lanolin 185.0 Borax 4.5 62.0 Distilled Water 1a Beeswax 300.0

<sup>13</sup> P. C.

<sup>&</sup>lt;sup>14</sup> Bennett, Chem. Formulary, Vol. 3, p. 109 (1936).

<sup>&</sup>lt;sup>15</sup> Ibid., Vol. 2, p. 371 (1935).

<sup>1. 1</sup>a Bennett, Chem. Formulary, Vol. 4, p. 51 (1939).

Pharmagel-A

#### MINERAL OIL EMULSIONS

# FORMULA No. 1. 8. g. Alcohol

60

CC.

Tartaric Acid Syrup Vanillin	0.6 100. 0.04	g. cc.	Water, To make Heavy Liquid Petrolatum <sup>2</sup>	500 500	cc.
, dillim		Ū	A No. 2.		-
Gelatin, Alkali Treate	ed.		Syrup	100	cc.
(Pharmagel B)	5	g.	Alcohol	60	cc.
Agar	2.5 - 5.0	g.	Water To Make	500	cc.
Sodium Bicarbonate	5	g.	Mineral Oil <sup>3</sup>	500	cc.
Vanillin	0.04	g.			

Dissolve the gelatin and agar in 100 cc of water by the aid of heat, cool somewhat, then add the syrup, the vanillin dissolved in the alcohol, the sodium bicarbonate and finally enough water to make the solution measure 500 cc., add the mineral oil, mix intimately by vigorous agitation and then pass through the homogenizer several times until thoroughly emulsified. Place the emulsion in a closed container and allow to stand over night whence it will be found to be set to a soft gel due to the agar. This is now passed in the cold through the homogenizer again, which will accomplish a mechanical dispersion providing a fluid emulsion which will not again set to form a gel.<sup>3</sup>

#### FORMULA No. 3.

Gelatin (0.6% Porkski	n,	Water, To make	440.0	сс
250 Bloom)	6.0	Vanillin	0.035	g.
Tartaric Acid or		Alcohol	60.0	cc
Hydrochloric Acid	0.450	Heavy Mineral Oil	500.0	cc
Syrup	100.0 cc.			

Add the gelatin and tartaric acid to about 300 cc. of cold water, allow to stand a few minutes, then heat until dissolved. Add the syrup and finally enough water to make 440 cc. (I). While still quite warm add II then III, and mix well avoid-

1800 cc.

ing the inclusion of air. Finally homogenize and bottle. Homogenization should be repeated if necessary until all the oil is completely dispersed.

Such an emulsion conforms to the New U. S. P. XI monograph for Emulsion of Liquid Petrolatum.<sup>4</sup>

#### FORMULA No. 4.

To obtain the best results, triturate sodium bicarbonate 10 grains, powdered tragacanth 20 and powdered acacia 60 grains to a smooth paste with glycerol 6 drams. Dissolve agar 17.5 grains in boiling water 2 fluid ounces, add this while hot to the mixture in the mortar while stirring. Then add liquid paraffin 3 ounces, 2–3 drams at a time. Triturate until cold, allow to stand for 1 hour, triturate again for 5 minutes and add water to complete 6 ounces. If an emulsifying apparatus worked by hand is available, a thick emulsion is obtained with agar 15, sodium bicarbonate 6 grains, liquid paraffin 3 ounces, tragacanth 5, acacia 5 grains, glycerol 2 drams, water to make 6 ounces.<sup>5</sup>

#### FORMULA No. 5.

Mineral Oil	gal.	Powdered Agar	1	1b.
Emulsone B or Gum		Citric Acid	2	oz.
Tragacanth	1b.			

Some sodium benzoate and aseptoform as preservative, and a small amount of vanillin and saccharin for flavoring purposes.<sup>6</sup>

FORMULA No. 6.

"A"

Gelatin 27 gr. Water

"B"

Mineral Oil 2000 cc.

<sup>5</sup> P. C.

<sup>&</sup>lt;sup>6</sup> Bennett, Chem. Formulary, Vol. 3, p. 101 (1936).

"C"

Agar	68 gr.	Tinc. Vanillin	4 fl. dr
Gum Arabic	110 gr.	Sodium Benzoate	2 dr.
Gum Tragacanth	110 gr.	Glycerin	4 fl. dr
Orange Oil	3 fl. dr.	Water	2000 cc.
Saccharin	10 gr.		

Make up "A" mixture and add "B" to same, using electric stirrer. Run mixture through colloid mill, using .010 gap setting.

Prepare "C" mixture and add to above. Run through colloid mill using .015 gap setting.

Add 960 grains phenolphthalein to each gallon of emulsion.<sup>7</sup>

#### FORMULA No. 8.

n 500.0 cc.	Tincture of Vanilla	8.0 g.
5.5 g.	Tincture of Lemon	2.0 cc.
120.0 g.	Cassia Oil	0.5 cc.
30.0 g.	Water, To make	1000.0 cc.
4.0 g.	,	
	5.5 g. 120.0 g. 30.0 g.	5.5 g. Tincture of Lemon 120.0 g. Cassia Oil 30.0 g. Water, To make

Mix the agar and the sugar with 300 cc. of boiling water and when they are dissolved strain the resulting solution and set it aside to cool. Triturate the powdered gums with the liquid petrolatum, then add the agar solution and whip the mixture with an egg beater. Finally add the tinctures and the oil and lastly enough water to make 1000 cc.<sup>8</sup>

#### FORMULA No. 9.

Powdered Tragacanth Moldex (Preservative)	5.0 1.0	Distilled Water Mucilage of Gum Acacia	304.5 30.0
Alcohol	10.0	Liquid Paraffin Medicinal	500.0
Glycerin	150.0		

If it is desired to include phenolphthalein in the emulsion, 10 of phenolphthalein is added in the place of an equal amount of water in the above formula.

<sup>&</sup>lt;sup>7</sup> Ibid., Vol. 1, p. 379 (1933).

<sup>8</sup> Ibid.

<sup>&</sup>lt;sup>9</sup> Bennett, Chem. Formulary, Vol. 1, p. 380 (1933).

#### FORMULA No. 10.

Liquid Petrolatum	500 cc.	Vanillin	0.035 g.
Acacia	125 g.	Alcohol	60 cc.
Syrup	100 cc.	Distilled Water To make	1000 cc.

Mix the liquid petrolatum with the powdered acacia in a dry mortar, add 250 cc. of distilled water all at once and emulsify the mixture. Then add, in divided portions and triturating after each addition, a mixture of the syrup, 50 cc. of distilled water and the vanillin, dissolved in the alcohol. Finally add sufficient distilled water to make the product measure 1000 cc.<sup>10</sup>

#### LINIMENTS

#### FORMULA No. 1.

Wintergreen Oil	60 cc.	Hydrous Lanolin	65 g.
Menthol	19 g.	Water	210 cc
Saponin	1 g.	Chloroform	10 g.

Dissolve the saponin in water. Mix wintergreen and menthol, add chloroform. Bring the two mixtures together and stir in the lanolin.<sup>11</sup>

#### FORMULA No. 2.

Raisin Seed Oil Methyl Salicylate		Diglycol Stearate Water 112	15 g 61 g
	Formula	No. 3.	

Avocado Oil	10 g.	Diglycol Stearate	22 g
Methyl Salicylate	15 g.	Water	60 g.

Heat diglycol stearate with oil at 70°C., until dissolved. While mixing add warm methyl salicylate and then pour mixture slowly with rapid mixing into water heated to 70°C.<sup>12</sup>

<sup>&</sup>lt;sup>10</sup> Ibid., Vol. 3, p. 101 (1936).

<sup>&</sup>lt;sup>11</sup> Bennett, Chem. Formulary, Vol. 2, p. 360 (1935).

<sup>11</sup>a Ibid., Vol. 5, p. 70 (1941).

<sup>12</sup> Ibid.

#### FORMULA No. 4.

Camphor, Powdered Turpentine	3 26		Blendene	10	lb.
I di pentine	20	ID.			

Mix the above until dissolved. Put in a pot fitted with a high speed agitator and while mixing rapidly add the following which has been dissolved previously:

Ammonium Chloride 7½ 1b. Water 28 gal. Stronger Ammonia Water 33/4 lb.

Continue mixing for about 15 minutes and bottle.13

#### FORMULA NO. 5.

Ammonium Carbonate	30 g.	Camphor	30 g.
Water	240 cc.	Alcohol	30 cc.
Castile Soap Hot Water	24 g. 480 cc.	Turpentine	q.s.

Dissolve the ammonium carbonate in 240 cc. of water. Dissolve the soap in the hot water, then mix the two solutions. Dissolve the camphor in the alcohol and add to the first mixture; shake well. Now add the oil of turpentine in sufficient quantity to make a creamy emulsion, gradually adding and constantly shaking.14

#### CASTOR OIL EMULSION

#### FORMULA NO. 1.

Castor Oil	2 oz.	Saccharin	4 gr
Powdered Acacia	6 dr.	Glycerin	3 dr
Peppermint Oil	3 min.	Cacao	2 dr
Powdered Tragacanth	16 gr.	Water To make	4 oz

A mixture of the cacao in the glycerin and water is made by poiling for five minutes. A mucilage of the acacia is made with 4 drams of the cacao mixture and the oils are gradually incorporated by trituration until emulsified. Then the remainder of the cacao mixture is added.1

<sup>18</sup> Ibid., Vol. 2, p. 358 (1935). 14 Ibid., Vol. 1, p. 383 (1933).

<sup>&</sup>lt;sup>1</sup> Bennett, Chem. Formulary, Vol. 2, p. 371 (1935).

#### FORMULA No. 2.

Sodium Hydroxide	9.8 gr.	Sodium Benzoate	100.5 gr.
Water	25.0 oz.	Triethanolamine	288.0 min.
Castor Oil	6 lb. 5.0 oz.		

Dissolve the sodium hydroxide in water, add sodium benzoate and add triethanolamine.

Mix with oil and run through colloid mill. For flavor use saccharine (water soluble) vanilla and lemon extract.2

#### FORMULA No. 3.

Aluminum hydroxide gel (3 per cent solids), 400 g.; castor oil, 700 g.; water, 50 g.; flavor and sweetening to suit.

The quantities of water, gel and castor oil are placed together, flavoring matter and sweetening having been previously added to the castor oil.

The whole is then beaten together mechanically, forming a thick, creamy mass which is more or less stable according to the amount of mixing.

To prepare the completed product for internal administration, this cream is usually passed through a homogenizer or similar suitable emulsifying apparatus. The final product is a finely dispersed, stable and white emulsion containing approximately 60 per cent castor oil combined with approximately 1 per cent of aluminum hydroxide on a dry basis.<sup>8</sup>

#### FORMULA No. 4.

Pharmagel-A	8 g.	Alcohol	60	cc.
Tartaric Acid	0.6 g.	Water, To make	500	cc.
Syrup	50 cc.	Aromatic Castor Oil N.F.	. 500	cc.

In making an emulsion of castor oil, the oil should be warmed to above 50°C. to render it more fluid and then mixed.4

<sup>&</sup>lt;sup>2</sup> Ibid., Vol. 1, p. 379 (1933). <sup>3</sup> Ibid., Vol. 4, p. 81 (1939).

<sup>&</sup>lt;sup>4</sup> Amer. J. Pharm. III 4 (1939).

#### WATER DISPERSABLE ANTISEPTIC OIL

WITTER DISTERSTED IN TISEL TO OIL				
Diglycol Laurate Coal Tar Distillate	20 <b>3</b>	Mineral Oil 1		77
A	ANTISEPT	IC LIQUID		
Oleic Acid Ti-tree Oil Coconut Fatty Acid	7 3 12	Triethanolamine Glycerin Water <sup>2</sup>		10 <b>2</b> 66
MERCU	RIC NITR	ATE OINTMENT		
Mercuric Nitrate Nitric Acid Distilled Water	11.34 1.35 32.31	Beeswax U.S.P. Cholesterol White Petrolatum		5.00 1.50 48.50
Mix 11.34 g. of	finely pow	vdered mercuric	nitrate	with

1 cc. of water, preferably in a mortar, and add 1.35 g. of nitric acid, accurately weighed. Triturate in the mortar until solution is effected and add 31.31 g. of water, slowly and with constant stirring.

Melt the white petrolatum, the cholesterol finely powdered and the white wax in a suitable dish. Continue the heat until the temperature of the mixture is raised to 80°C., and the cholesterol has completely dissolved. Stir the mixture until it congeals. By trituration, slowly incorporate the aqueous solution of mercuric nitrate into the ointment base. Care should be taken to avoid contact with metallic instruments or containers 3

#### POISON IVY LOTION

Ammonium Oleate	5.0	De-Odorized Kerosene	77.4
White Mineral Oil	10.5	Eugenol	5.0
Menthol	0.1	Carbon Tetrachloride	2.0

Dissolve the menthol in the eugenol, add the mineral oil and the carbon tetrachloride and then dissolve the ammonium oleate in the mixture. Add this to the kerosene.4

<sup>&</sup>lt;sup>1</sup> Bennett, Chem. Formulary, Vol. 4, p. 59 (1939).

<sup>&</sup>lt;sup>3</sup> Ibid., Vol. 5, p. 69 (1941). <sup>4</sup> Ibid., Vol. 2, p. 365 (1935).

3 6 7 7 7	~	
Medicinal	F.m:	11.51.0115

339

#### PSORIASIS LOTION

Mace Oil	10	Rose Water	5
Olive Oil	10	Lecithin	50
Ammonia	15	Chloroform	1/2
Essence of Rosemary	5	Perfume to suit 5	•

#### COLCHICINE EMULSION

Stearic Acid Morpholine		Lanolin Colchicine <sup>6</sup>	8.00 g. 0.12 g.
MOI phonne		Colcincing	U.12 5.
Tap Water	20.00 cc.		

#### SULFANILAMIDE EMULSION

Sulfanilamide	175 gr.	Calcium Hydroxide 7	
Cod Liver Oil	4 fl. oz.	(Saturated Solution)	8 fl. oz.
Oleic Acid U.S.P.	36 min.		

#### HAY FEVER OINTMENT

Lanolin Anhydrous	50	Menthol	1/2
Yellow Petrolatum	25	Epinephrin Solution 1-1000	2
Ethyl Amino Benzoate	5	Distilled Water	23

- (1) Triturate ethyl amino benzoate and menthol with a portion of the yellow petrolatum until smooth. Gradually add the remainder of the petrolatum and the lanolin.
- (2) Mix epinephrin solution with distilled water and add this aqueous solution slowly under trituration to No. 1 and mix until homogeneous.8

#### DISINFECTANT EMULSION

Orthophenyl Phenol	10-40%	Sulphonated	Castor
Terpene Ether	15%	Oil 9	To make 100%

<sup>&</sup>lt;sup>5</sup> Ibid., Vol. 1, p. 142 (1933).

<sup>6</sup> P. C.

<sup>&</sup>lt;sup>7</sup> Drug & Cosmetic Industry 50, 146 (1942).

<sup>8</sup> Bennett, Chem. Formulary, Vol. 1, p. 386 (1933).

<sup>9</sup> U. S. 2,289,476.

#### CHAPTER XIV

# PAINT EMULSIONS

#### FORMULA NO. 1.

Hydrated Lime	43.0	Mica	5.0
Hydraulic Cement	19.5	Gum Arabic	1.6
Talc	12.0	Gum Karaya	0.5
Metronite	11.5	Irish Moss	0.1
Salt	6.5	Calcium Stearate	0.3

Add water to suit before use.1

#### FORMULA No. 2.

The following mixtures are prepared separately

1.	Potato Starch Water	10 30	Water Caustic Soda (10° Bé.)	20 10
	Caustic Soda (10° Bé.)	10	3. Water	150
۷.	Casein	0	Linseed Oil Varnish	80

These three mixtures can be stirred together into a smooth emulsion which will remain stable for 6-8 months.<sup>2</sup>

#### FORMULA No. 3.

1. Casein	30	2. Formaldehyde (40%)	2
Water	150	Water	5
Borax	3.5	3. Rosin	15
Phenol	1	Linseed Oil Varnish	15
		White Spirit	10

<sup>Schloz, Can. 317,827 (1931).
Hadert, Casein & Its Uses.</sup> 

The casein is soaked in hot water, and the borax and later the phenol are added. After standing for 24 hours, the ingredients of group 2 are mixed in, followed by the ingredients of group 3 which had been mixed hot previously. Since the pigments are more easily wetted in oil than in water, it is advisable to grind them with the oil binder 3, before this is emulsified with the casein solution. The amount of pigment averages about 6 to 8 times the amount of the binder. Casein paints thus made excel by their good brushability and increased water resistance.<sup>3</sup>

#### FORMULA No. 4.

Dehydrated Castor Oil	33.41	Phenol	0.06
Lithopone	39.81	Ammonium Hydroxide	23.41
Casein	3.31		

Mix well until uniform; dilute before using.4

#### FORMULA No. 5.

Shellac	30	Water	65.5
Ammonium Bisulphate	4.5		

Mix until dissolved and then add

Linseed Oil 30

Emulsify with electric mixer and add

Lithopone 5

380

#### FORMULA No. 6.

Water Ground Mica	Casein, Muriatic (80 Mesh)	1 lb.
Lithopone or Titanox	Sodium Fluoride	2 oz.

#### FORMULA No. 7.

Montan Wax, Double		Potash, Caustic	0.8
Bleached	7.0	Water Soluble Dye	2.0
Potassium Oleate	3.0	Water	87.2

<sup>8</sup> Ibid

<sup>4</sup> Bennett, Chem. Formulary, Vol. 5, p. 341.

<sup>&</sup>lt;sup>5</sup> Ibid.

<sup>6</sup> P. C.

The potash, the soap and the dye are dissolved in the boiling water. The boiling solution is then slowly poured into the wax melt adding it in small portions at a time. Care must be taken that a drop in temperature does not cause a separation. It is essential that the mixture be stirred constantly while cooling.<sup>7</sup>

#### FORMULA No. 8.

Crude wool fat, refined tung oil and rosin, are melted up in the respective proportions of 360: 40: 250, the melt being incorporated with 43 parts of ammonia solution, 100 parts alcohol and 1207 parts water and the resulting emulsion agitated till cold. The product at this stage, a viscous, yellowish-white emulsion, may be directly employed as a paint. An example of a quick-drying paint comprises 1000 parts of above emulsion, 80 parts chrome oxide, 150 parts titanium white and 15 parts of a 33 per cent solution of a cobalt-lead-manganese drier.8

#### FORMULA No. 9.

Double Boiled Oil, with Driers	50	Sodium Silicate	5
Water	45	Pigment	50

The oil, which may be diluted if required with 120°F. flash white spirit, should be added to the aqueous phase in a slow stream with rapid and vigorous stirring.9

#### FORMULA No. 10.

1. Sodium Silicate	10	Zinc Dimethyldithio	
Ammonia	10	Carbamate	1/2
Water	10	2. 60% Latex	100
Zinc Öxide	5	Whiting	200
Sulphur	3	Spindle Oil	60
		Glue	5

<sup>&</sup>lt;sup>7</sup> Ibid., Vol. 4, p. 327 (1939).

<sup>8</sup> P. C

<sup>9</sup> Bennett, Chem. Formulary, Vol. 1, p. 269 (1933).

The two solutions are made separately as indicated, and mixed. The more alkaline varieties of sodium silicate cause precipitation of latex by reason of hydrolysis. If, however, ammonia be added to the solution this increases the OH ion concentration and prevents splitting of the silicate, so that the latex is thickened and rendered stable.<sup>10</sup>

#### FORMULA No. 11.

A. Potato Starch	10	Caustic Soda (10° Bé.)	10
Cold Water	30	,	

Mix the starch with cold water and add the caustic slowly in a thin stream till a transparent thick liquid is obtained.

B. 90-Mesh Lactic Casein	6	Caustic Soda (20° Bé.)	10
Water	20	•	

Soak the casein in the warm water, not over 130°F., and add the caustic whilst stirring.

C. Medium Congo Copal	20		
Linseed Oil	50	Linseed Oil Varnish	80
White Spirit	30	Water	150
Manganese (as Resinate)	0.1		

Grind in the required amount of pigment with the oil varnish and then stir in the water. Run the three solutions together through a Hurrel Homogenizer and the resulting emulsion will be stable for a year. If for export to a hot country, it is advisable to add a little preservative, e.g., metachlor-paracresol.<sup>11</sup>

#### FORMULA No. 12.

Trihydroxyethylamine		Varnish	16.0
Linoleate	0.6	Naphtha	4.0
Glue	10.0	Sodium Ortho Phenyl	
Water	32.0	Phenate 12	0.1

<sup>10</sup> Ibid.

<sup>11</sup> P. C

<sup>12</sup> Ibid., Vol. 1, p. 270 (1933).

#### FORMULA No. 13.

Beeswax	70	Shellac	10
Japan Wax	44	Potassium Carbonate	<b>3</b> 0
Montan Wax	25	Water	730

Boil together and mix well until emulsified. Cool and add with stirring

Alcohol		40	Trioxymethylene 13	50
	For	MULA	No. 14.	
Lithopone Asbestine Mica Vehicle Water	60.0 20.0 8.0	1b. 1b. 1b.	Dowicide "B" Sodium Alginate Raw Chinawood Oil Cobalt Drier 6% Wetting Agent	0.45 lb. 1.0 lb. 9.0 lb. 0.48 oz. 1.68 oz.

Place the water (preferably from the hot water tap at approximately 100°F.) in the mixer. Add the Dowicide "B" and stir until dissolved. Add the sodium alginate and stir for a full 20 minutes or until completely dispersed. Add wetting agent and then the chinawood oil (with the drier added to the oil before adding to the water mixture). Stir the oil and alginate mixture for 10 minutes and then add pigment and filler. Stir for one hour. Remove from mixer and put into containers.<sup>14</sup>

#### FORMULA No. 15.

Sodium Silicate	2 lb.	Pale Boiled Linseed Oil	¼ 1b.
Soap	½ lb.	Water (Boiling)	1 gal.

Mix very thoroughly. A little alum may be added to increase water resistance.<sup>15</sup>

#### FORMULA No. 16.

Oiticica Oil	120	Manganese	Dioxide
Lead Oxide	6		

<sup>18</sup> Bennett, Chem. Formulary, Vol. 4, p. 300 (1939).

<sup>14</sup> Ibid., p. 297.

<sup>15</sup> P. C.

# Heat to 250°C. and then reduce to 200°C. and add

Potassium Silicate		Water	sufficient
Milk of Lime	16		

Agitate violently until cool.1

#### RESIN OIL EMULSION

	Soluti	on "A"	
Ester Gum Bodied Linseed Oil Pine Oil Mineral Spirits	62 50 21 62	Co. Linoleate Drier (6% C Pb. Linoleate Drier (16% Oleic Acid	
	Soluti	on "B"	
Powdered Casein Preservative (Moldex)	27 3⁄8	Emulsifier (2 Amino-2 Methyl-1 Propanol) Water	15 536

Mix the resin, cut in the mineral spirits, with the bodied linseed oil, pine oil, driers, and oleic acid.

Place the casein in mixer, add the water slowly, stirring so that casein is wetted, add the preservative and emulsifier and continue stirring for several minutes.

Add Solution "A" slowly to Solution "B" with constant high speed agitation until emulsification is complete.1

#### SEMI-PASTE PAINTS

# FORMULA No. 1. Albalith-335, 332 or 836 790 Ester Gum Oil Emulsion 622 FORMULA No. 2. Cryptone ZS-830 376 Ester Gum Oil Emulsion 625 China Clay 230

These semi-pastes are designed to be thinned 2-1 with water for brush application.

The illustrative formulae of the emulsion and semi-pastes given here can be modified in many ways, but in making

<sup>16</sup> Ibid., Vol. 3, p. 52 (1936).

substitutions, or if the percentages of the ingredients are changed, the new system must be balanced or the breaking of the emulsion may follow. This adds a variable that is different from the type of problem that the paint manufacturer encounters in his everyday paint making and introduces elements that cause slow progress to be made in investigational work 2

#### "PICCOLYTE" OIL EMULSION (V-338)

#### SOLUTION "A" 30.5% Piccolyte S-100 Resin 27.2% Co. Linoleate Drier 22.0% (6% Co.) .5% Lithographic Oil #8 9.3% Pb. Linoleate Drier Pine Oil 27.2% (16% Pb.) 1.9% Mineral Spirits Oleic Acid 11.9% SOLUTION "B" 69.5%

Casein B-5	5.2%	2 Amino-2	Methyl-1	
Moldex or Dowicide "A"	.1%	Propanol	-	2.9%
	•	Water		91.8%

Dissolve the Piccolyte resin in the mineral spirits and mix with the lithographic oil No. 8, pine oil, driers, and oleic acid.

Place the casein and preservative in a pony or other type mixer, add the water slowly, stirring so that casein and preservative are wetted, add the 2 Amino-2 Methyl-1 Propanol, and continue the stirring for several minutes.

Add Solution "A" slowly to Solution "B" with constant agitation until emulsification is complete.3

#### "PICCOLYTE" OIL EMULSION SEMI-PASTE

	INTERIOR	
•	Pigment 54.5%	
Cryptone ZS-830	62% China Clay	38%
	Vehicle 45.5%	
Piccolyte Emulsion V-338	93.1% Water	6.9%

The pigment, emulsion and water should be mixed together and then milled on a roller or buhrstone mill at a loose setting.<sup>4</sup>

<sup>1-4</sup> inc.: N. J. Zinc Co., New York, N. Y.

#### CHAPTER XV

# PAPER PROCESSING EMULSIONS

# PAPER SIZINGS FORMULA No. 1. Paraffin Wax 75 Soap 10 Carnauba Wax 25 Water 1 To suit FORMULA No. 2. Japan Wax 100 Water 40 Soap 10

Boil and stir until homogeneous. This is diluted with boiling water and stirred before use.<sup>2</sup>

## FORMULA No. 3.

Rosin	70	Beeswax	2
Soda Ash	7	Water	21

Boil together until a sample solidifies on cooling. This may be shipped solid and is dispersed in hot water when needed.<sup>3</sup>

#### FORMULA No. 4.

700 parts of resin are melted with 200 parts of turpentine oil of high boiling point, e.g., that known commercially as Yarmor pine oil, and are stirred into a solution of a caseinate-

<sup>&</sup>lt;sup>1</sup> P. C.

<sup>&</sup>lt;sup>2</sup> Ibid., Vol. 1, p. 476 (1933).

<sup>&</sup>lt;sup>3</sup> Ibid., p. 475.

resin soap emulsifying medium in 1,000 parts of water preferably using a steam jet or high-speed rotary stirrer. The resin soap is made by adding 100 parts of commercial caseinate powder to 80 parts of powdered resin and a 10 per cent solution of ammonia is added with mild heating to the mixture which is kneaded till saponification is completed and the mixture of swollen ammonium caseinate and ammonium resinate has reached its highest viscosity.<sup>4</sup>

#### FORMULA No. 5.

Ten parts of soy bean protein are treated with 0.7 part of sodium carbonate by heating to 60°C. under mild agitation with 60 parts of water. To this is added five parts of ammonium oleate for other suitable emulsifying agent, such as turkey red oil, gums like gum arabic, sodium stearate, etc. To this mixture, 40 parts of molten paraffin is slowly run in, under agitation. The mixture of casein, emulsifier, and paraffin is stirred with a high speed stirrer or other suitable equipment. The time of mixing is usually approximately 15 minutes, the mixture then being diluted with about 1000 parts of water at approximately 60°C. These temperatures can be adjusted, depending upon the melting point of the paraffin used. The emulsion can likewise be made through the use of a colloid mill or other suitable equipment.<sup>5</sup>

	FORMULA No. 6.	
Rosin Colloidal Clay	85 Water 15 Caustic Soc	100 da <sup>6</sup> 2
	FORMULA No. 7.	
Petroleum Resin Colloidal Clay Casein	37 Caustic So 18 Water <sup>7</sup> 1½	da 1 To suit

<sup>4</sup> Ibid., Vol. 2, p. 345 (1936).

<sup>5</sup> Ibid.

<sup>&</sup>lt;sup>6</sup> Manson, U. S. 1,990,457 (1935).

<sup>&</sup>lt;sup>7</sup> Paper Makers' Assn., London, Eng. (1942).

#### WATERPROOFING EMULSIONS FOR PAPER

#### FORMULA No. 1.

Paraffin Wax	33	Glue	1
Silicate of Soda	4	Water	60
Alum	2		

The silicate of soda used in the above formula is a commercial form of syrupy consistency containing about 50 per cent of water. This silicate of soda syrup is mixed with 65 per cent of the total water. The remaining 35 per cent of the total water is used to dissolve the alum. The glue is added to the diluted silicate of soda. The wax composition is prepared by first melting the wax, adding the silicate of soda solution containing the glue and agitating, finally adding the alum solution with agitation. The temperature at which the composition is prepared is about 170°F.¹

#### FORMULA No. 2.

Paraffin Wax	32.50	Alum	2.75
Montan Wax	0.85	Glue	0.40
Silicate of Soda	5.00	Water	58.50

In this case the silicate of soda specified is a syrup containing about 50 per cent of water. This syrup is diluted with 65 per cent of the total water and the alum is dissolved in the remainder of the water employed.

The glue is dissolved in the silicate of soda solution. It is desirable to add 1 or 2 per cent of phenol, based on the amount of glue, in order to preserve the latter. The waxes are melted together and the silicate of soda solution containing the glue is added thereto with thorough agitation, the temperature being about 170°F. The alum solution is then added and agitation continued for a short time.<sup>2</sup>

<sup>&</sup>lt;sup>1</sup> Bennett, Chem. Formulary, Vol. 2, p. 350 (1935).

<sup>2</sup> Thid.

	Paper Processing Emulsions	351
	FORMULA No. 3.	
ı	31.00 Alum 7.00 Water <sup>3</sup> 2.65	1.35 58.00
	FORMULA No. 4.	

22.5 Water 4 Paraffin (130°F.) 74.5 Trihydroxyethylamine Stearate 3.0

FORMULA No. 5.

Water 5 Paraffin Wax 50 980 Ammonium Stearate 70

FORMULA No. 6.

Stearic Acid Trihydroxyethylamine 1/2 41/2 Water 100 Stearate

Boil and mix until smooth; pour into this slowly while stirring vigorously

Paraffin Wax (Heated to 90-100°C.) 30

Stir until cool.

Paraffin Wax

Montan Wax

Silicate of Soda

Use 1 part of above emulsion to 5-10 parts of warm water.6

## FORMITIA NO. 7.

Latex (60%)	50	Sodium Silicate	5
Paraffin Emulsion (50%)	30	Water	30

The paraffin emulsion is diluted with the water and the silicate stirred in. The latex is added last and stirred until homogeneous.7

<sup>8</sup> Ibid.

<sup>4</sup> Ibid., p. 349.

<sup>&</sup>lt;sup>5</sup> Kniffler, U. S. 1,266,955 (1918).

<sup>&</sup>lt;sup>6</sup> Bennett, Chem. Formulary, Vol. 3, p. 255 (1936).

<sup>&</sup>lt;sup>7</sup> Ibid., Vol. 2, p. 348 (1935).

#### FORMULA No. 8.

Stearic Acid	4 lb.	Triphenyl Phosphate	8 lb.
Japan Wax	5 lb.	Dibutyl Phthalate	1 lb.

Heat to 90°C. and add

Water Shellac (40%) 56 lb. Triethanolamine 2 lb.

Cool to 70°C. and add successively with vigorous stirring

Ammonia (28%)	1 qt.	Water	3 gal.
Water	3 gal.	Latex + 4% Sul	phur 3 lb.
Ammonia (28%)	3 qt.	Water 8	To make 28 gal.

#### EMULSION FOR HIGH LUSTRE PAPER

#### FORMULA No. 1.

Marseilles Soap	2.0	Water, Free of Calcium 9	40.0
Montan Wax	6.5		

#### FORMULA No. 2.

Yellow Laundry Soap	7 lb.	Water	12½ gal.
Carnauba Wax	50 lb.		

Boil with live steam till thoroughly emulsified (from 3-4 hours).

Cool to 35°C. and add

Ammonia (28°) 2 lb. Cold Water to make 50 gal.

The emulsion should be allowed to stand for at least 24 hours before use as it seems to improve with age. 10

#### FORMULA No. 3.

Dry Castile Soap	12 lb.	Boil till dissolved, then add	
Water	25 gal.	Carnauba Wax	75 lb.

Boil three hours; cool to 100°F. and add 3 pints 26° ammonia. Make up to 75 gallons with water. This emulsion is added to the casein coating mixture.<sup>11</sup>

<sup>&</sup>lt;sup>8</sup> Ibid., Vol. 3, p. 255 (1936).

<sup>9</sup> P. C.

<sup>10</sup> Ibid., Vol. 3, p. 254 (1936).

<sup>&</sup>lt;sup>11</sup> Bennett, Chem. Formulary, Vol. 2, p. 348 (1939).

#### DE-INKING PRINTED PAPER

For separating the cellulosic fibers of filled and printed paper, the paper is beaten in a solution containing sodium hydroxide, sodium metasilicate or sodium phosphate in such proportion as to give the solution a pH of 9.0–12.6 until the paper has been substantially disintegrated, and an emulsifying agent such as a soap or sulphonated oil is then added to the resulting aqueous suspension and the beating is continued for a short time. The material is filtered through a sieve fine enough to retain the cellulosic fibers but coarse enough to pass the fillers and ink particles.<sup>12</sup>

<sup>12</sup> Ibid., Vol. 2, p. 346 (1935).

## CHAPTER XVI

## POLISH EMULSIONS

#### AUTOMOBILE POLISHES

## FORMULA No. 1.

1. Acetic Acid (80%)		Rosin Oil	4
or Lactic Acid	4	Camphor Oil, Viscous	2
Water		3. Neuburger Chalk or	
2. Spindle Oil, Refined	9	Tripoli. Finest 1	7

To solution 1 add the mixture 2. Stir until homogeneous, and stir in 3.

## FORMULA No. 2.

a. Ammonium Linoleate	20	c. Water	15
Turpentine	15	d. Whiting	45
b. Alcohol	5		

Dissolve a, add b, c, and ultimately d. Mix thoroughly.<sup>2</sup>

## FORMULA No. 3.

A. Carnauba Wax	30	B. Water	70
Glyco Wax B	20	Borax	10
Naphtha or Varnolene	68		
Turpentine	17		

Melt "A" together but do not heat above the boiling point of water. Meanwhile dissolve "B" while heating to a boil.

Run "A" into "B" slowly while stirring vigorously.3

<sup>&</sup>lt;sup>1</sup> Bennett, Chem. Formulary, Vol. 4, p. 403 (1939).

<sup>&</sup>lt;sup>2</sup> Ibid., p. 437.

<sup>8</sup> Ibid., Vol. 1, p. 418 (1933).

## FORMULA No. 4.

Carnauba Wax	120	Oleic Acid	3
Kerosene	50	Benzaldehyde	6
Stearic Acid	15	Triethanolamine	8

Melt the carnauba wax, stearic acid and oleic acid. Remove from fire and add the kerosene and the benzaldehyde. Stir thoroughly. Now add a hot solution (80-85°C.) consisting of 240 parts of water and the triethanolamine. Stir well until a smooth emulsion is formed, then add sufficient hot water to make 825 parts. Continue stirring until nearly cool then hottle.4

## FORMULA No. 5.

8 Paraffin Wax Carnauha Wax 4 Montan Wax

These are saponified in a hot solution of:

3 Water Potash 40

Replace any evaporation with additional warm water. There is then added to this 20 parts of turpentine.<sup>5</sup>

## FORMULA NO. 6.

a. Carnauba Wax, Gray	10	b. Black Soap	11/2
Montan Wax, Bleached	3	Water	10
Japan Wax	5	c. Solvent (Turpentine	
Scale Wax (50-52°C.)	2	White Spirit, etc.)	60

Melt a at 95–100°C., prepare the hot soap solution b.

Add c with good agitation, and stir until it begins to go heavy (at about 48°C.).6

#### FORMULA NO. 7.

a. Carnauba Wax, Gray	15	c. Solvent (Turpentine, White
Beeswax or Japan Wax	5	Spirit, or a Mixture of both
Glycowax	5	and 10 parts of Kerosene) 30
b. Kaolin or Floated Chalk	30-35	d. Black Soap
		Water 7–10

<sup>&</sup>lt;sup>4</sup> Ibid., Vol. 2, p. 421 (1935). <sup>5</sup> Ibid., Vol. 1, p. 430 (1933).

<sup>&</sup>lt;sup>6</sup> Bennett, Chem. Formulary, Vol. 4, p. 405 (1939).

## FORMULA No. 14.

To 3½ gal. of pale blown castor oil, add ¾ gal. of orthodichlorbenzol. This is mixture No. 1. To 15 gal. of water, add 11 gal. of neutral pale mineral oil and ¾ gal. of ammonia, which has been previously made up of one part of ammonia of 26° Bé. and 4 parts of water. This is mixture No. 2. Mixture No. 1 and mixture No. 2 are combined and agitated for about 5 minutes. Three and one-half gallons of special petroleum spirit is added and the whole mass is now stirred about 10 minutes. It is then run through a colloid mill and is ready for use. Alternately, all of the ingredients may be mixed in a single batch and passed through the colloid mill, which breaks up the particles to a fine degree. This obviates preparing separate mixtures.¹⁴

## FORMULA No. 15.

Kieselguhr Paraffin Wax Methylated Spirit	8 8 2	Glycerin Water Gum Tragacanth <sup>15</sup>	2 40 ½
	Formula	No. 16.	
Kieselguhr (Levigated) Silica (Levigated) Yellow Ochre Red Ochre	11 9 1 1/10	Kerosene Soft Paraffin Wax Powdered Soap 18	16 2 1
	Formula	No. 17.	
Mineral Oil, White Beeswax Carbon Tetrachloride Kieselguhr	35 5 10 3	Oleic Acid Potash Carbonate Ammonia Water <sup>17</sup>	3 1/2 1/2 43

<sup>&</sup>lt;sup>14</sup> Tumbler, U. S. 1,969,387.

<sup>&</sup>lt;sup>15</sup> Bennett, Chem. Formulary, Vol. 3, p. 286 (1936).

<sup>&</sup>lt;sup>18</sup> Ibid., Vol. 1, p. 418 (1933).

<sup>&</sup>lt;sup>17</sup> Bennett, Chem. Formulary, Vol. 4, p. 404 (1939).

#### FORMULA No. 18.

<ul> <li>a. Montan Wax, Bleached</li></ul>	3.0	b. Potash Carbonate Borax Black Soap Water c. Kerosene	0.2
Carnauba Wax Residues	4.0		0.3
Yellow Beeswax	1.5		0.5
Japan Wax	1.5		35.0
Glycowax	1.0		10.0
		Turpentine	10.0

Melt a at about  $100^{\circ}$ C., and add b, of equal temperature, to it with very good agitation. Watch closely to prevent from foaming over (carbon dioxide formed!) and keep boiling until the saponification is finished and add c. Stir until cold.<sup>18</sup>

FORMULA NO. 19.	
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Naphtha	2½ pt.	Ammonia	oz.
Mineral Oil	2½ pt.	Water 19	pt.
Diglycol Laurate	4 pt.		

## FORMULA No. 20.

Oleic Acid	80	Tragacanth	6
Liquid Paraffin	250	Water, To make	1000
Potassium Hydroxide	16		

Mix the oleic acid with the paraffin and slowly add the potassium hydroxide, previously dissolved in 200.0 of water. Soak the tragacanth in 500 cc. of water until fully absorbed, then heat to boiling, and when cool stir into the above emulsion.

#### FORMULA No. 21.

Oleic Acid	2	1Ь.	Water	28	1b.
White Mineral Oil	12	1b.	Triethanolamine	13	oz.
Carnauba Wax	21/2	1b.	Infusorial Earth	100	lb.
Yellow Beeswax	2	1b.	Gum Tragacanth	4	1b.

Take the carnauba wax, beeswax, oleic acid and white mineral oil and melt at gentle heat. In another vessel heat to

<sup>&</sup>lt;sup>18</sup> Ibid., p. 405.

<sup>&</sup>lt;sup>19</sup> Ibid., Vol. 5, p. 534 (1941).

<sup>&</sup>lt;sup>20</sup> Ibid., Vol. 1, p. 418 (1933).

near boiling point the water and the triethanolamine. Add the oil-wax mixture to the water mixture in a slow stream under steady stirring and retain at near boiling temperature until a smooth emulsion has been formed. Then remove from fire and continue stirring until nearly cold. Now add to the emulsion the infusorial earth, mix well and let dry until all the water has been expelled. Then add the powdered gum tragacanth, mix thoroughly and sift. 8 to 12 ounces of the powder are sufficient to make 1 gallon of polish by merely adding sufficient water.<sup>21</sup>

	FORMULA	No. 22.	
a. Olein Mineral Oil Petroleum Turpentine Oil or White Spirit	20 cc.	<ul> <li>b. Alcohol         Ammonia (0.910)     </li> <li>c. Infusorial Earth <sup>22</sup></li> </ul>	6 cc. 6 cc. 10 g.
	Formula	No. 22a	
Olein, Distilled White Spirit	15 g.	Chalk, Neuburg, Fine Ammonia, 25%	45 g. 1 g.
(Lacquer Benzoline) Saponify this mixtu	90 g. re addin	g the ammonia with th	horough

Saponity this mixture adding the ammonia with thorough agitation.<sup>22a</sup>

	Formula	No. 23.	
Paraffin Oil Blown Castor Oil Kieselguhr China Clay, Colloidal	20.0 2.0 4.0 1.0	Tragacanth, Gum Sodium Benzoate Water <sup>23</sup>	0.6 0.2 72.2
	FORMULA	No. 24.	
Soap Kieselguhr	20 10	Mineral Oil Naphtha	5
Kaolin Iron Oxide	15	Water 24	4()

<sup>&</sup>lt;sup>21</sup> Ibid., Vol. 2, p. 421 (1935).

<sup>&</sup>lt;sup>22</sup> Bennett, Chem. Formulary, Vol. 3, p. 285 (1936).

<sup>&</sup>lt;sup>22a</sup> Ibid., Vol. 4, p. 404 (1939).

<sup>&</sup>lt;sup>23</sup> Ibid., p. 405.

<sup>&</sup>lt;sup>24</sup> Ibid., Vol. 4, p. 403 (1939).

## FORMULA No. 25.

Sulphonated Olive Oil (30%)	10	Amyl Alcohol	10
Mineral Oil	30	Carnauba Wax (Powdered)	5
Pumice Powder	5	Water	40

Mix the oils, wax and alcohol; add the water slowly with good stirring; mix in other ingredients.<sup>25</sup>

## Formula No. 26.

Turpentine	25	Iron Oxide	5
Naphtha	1,5	Mineral Oil	2
Oleic Acid	10	Water	10
Kaolin	15	Triethanolamine 26	3
Whiting	15		

## FORMULA No. 27.

Turpentine	381/2	Iron Oxide	5
Oleic Acid	15	Mineral Oil	5
Kaolin	20	Water	10
Kieselguhr	5	Potash, Caustic 27	11/2

#### FORMULA No. 28.

Water	55	Turpentine	15
Glue, Pale	6	Spindle Oil, Thin	14
Lactic Acid	3	Perfume (Pine Needle Oil,	
Sand, Finely Sifted,)		or similar oil) 28	To suit
"0000," or	7		
"Silver-Tripoli," or			
Kieselguhr			

#### FORMULA No. 29.

a. Soluble Oil	35	c. Soluble Oil	25
Talloil	30	Kerosene	15
b. Caustic Soda (38° Bé.)	8	d. Water	5-10
,		e Turkey Red Oil	5~ 7

Mix a, add b at 70°C., very slowly with agitation. Boil. When saponified, add c, and d with stirring (all hot); e may be added is desired.<sup>29</sup>

<sup>&</sup>lt;sup>25</sup> Ibid., Vol. 2, p. 421 (1935).

<sup>&</sup>lt;sup>26</sup> Ibid., Vol. 4, p. 403 (1939).

<sup>27</sup> Ibid.

<sup>&</sup>lt;sup>28</sup> Bennett, Chem. Formulary, Vol. 4, p. 403 (1939).

<sup>2°</sup> Ibid., p. 405.

#### FORMULA No. 30.

a. Oleic Acid	5.0	c. Kerosene	70.0
Rosin, In small pieces	6.0	Turkey Red Oil	8.0
Alcohol		d. Water	5-10.0
b. Caustic Soda (38° Bé.)	2.5		

Heat a—with the amount of alcohol desired. Mix well, and add b at about 70°C. Add c with continuous agitation. The be added if desired <sup>30</sup>

be added it desired.		•	
Wax Stearic Acid Potash, Caustic	Formula 25.0 2.5. 2.5	No. 31. Turpentine Water <sup>81</sup>	20.0 To make 100.0
	FORMULA	No. 32.	
Wax Stearic Acid Ammonia (0.88)	20.0 2.5 2.0	Triethanolamine Turpentine Water 32	1.5 25.0 To make 100.0

#### FORMULA No. 33.

White Mineral Oil	80	Triethanolamine	4
Deodorized Mineral Oil	9	Methanol	4
Stearic Acid	9	Water	120

Mix the white oil and mineral oil and add the stearic acid. Heat the mixture to about 140°F. at which time the stearic acid will dissolve to give a clear solution. In a separate container mix the triethanolamine, methanol and water and heat likewise to about 140°F. Then add the first mixture to this and stir vigorously until the emulsion is smooth. Continue to stir gently until cool.<sup>33</sup>

#### FORMULA No. 34.

a. White Oil	20	b. Water	100
a. Willie Oli	20	U. Water	100
Castor Oil	10	Neuburg Chalk	25
Solvent Naphtha	5	Kaolin, Finest	5
Glycerin	5	Bentonite	3
Turpentine	15		
Diglycol Laurate	21/		

<sup>30</sup> Ibid.

<sup>81</sup> Ibid.

<sup>32</sup> Ibid.

<sup>38</sup> Ibid., Vol. 2, p. 421 (1935).

Make mixtures a and b, and add a to b. Homogenize, if desired.<sup>34</sup>

FORMULA N	lo. 35.	
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White Spirit Mineral Oil		Glycerin	5 pt. 1 pt.
Turkey Red Oil Ammonia	4 pt. 1 oz.	Formaldehyde 35	8 oz.

#### FORMULA No. 36.

Fuller's	Earth	8	Bentonite

Mix the oils together first and add the abrasive powders, then the water, ammonia, glycerin, and formaldehyde; stir rapidly until a smooth mixture is obtained.<sup>36</sup>

#### FORMULA No. 37.

a. Yellow Beeswax or		b. Potash Carbonate	3.0
Montan Wax, Refined	36.0	Water	140.0
		c. Turpentine or Substitute	10.5

Melt a carefully; add slowly a *boiling* solution b, boil until the saponification is completed. Cool, thin with c.<sup>37</sup>

#### FORMULA No. 37a

Yellow Wax	10	Soft Soap	1/2
Air-Floated Tripoli	18	Water	21/2
White Spirit	19		-/-

Melt the wax in a double pan and add the powder slowly; keep stirring while slowly adding the white spirit. Dissolve the soft soap in the water and add to the mix with constant stirring. On cooling this forms a soft paste.<sup>87a</sup>

#### FORMULA No. 38.

Yellow Wax	20	Turpentine Substitute	40
Commercial Silica, Very		Soft Soap	1
Finely Powdered	40	Water	5

<sup>34</sup> Bennett, Chem. Formulary, Vol. 4, p. 405 (1939).

<sup>35</sup> Ibid., Vol. 3, p. 285 (1936).

<sup>36</sup> Ibid., p. 286.

<sup>&</sup>lt;sup>87</sup> Ibid., Vol. 4, p. 405 (1939).

<sup>&</sup>lt;sup>37a</sup> Bennett, Chem, Formulary, Vol. 3, p. 285 (1936).

Melt the wax and incorporate the powder, slowly adding the turpentine substitute, finally stir in the soap, previously dissolved in the water. Some may prefer it to be without the soap, but experience shows it to be worth its slight softening effect in yielding a higher and better polish. The paste may be tinted with ferric oxide.<sup>38</sup>

## FLOOR POLISHES

	Formula No. 1.	
"Aquamel"	15 Water	225
Carnauba Wax	25	

Heat the wax and "Aquamel" together till a clear jelly is obtained. Then add the water which must be as near boiling as possible. Stir thoroughly till cool.

#### FORMULA No. 2.

Carnauba Wax	10.0	Refined Shellac	1.4
Triethanolamine	1.4	Ammonium Hydroxide	0.2
Oleic Acid	1.4	Water to Make 2	
Borax	1.0		

## FORMULA No. 3a

A stable emulsified floor wax can be obtained with 40% of a mixture of 1 part of beeswax, 1 part of paraffin, 1% of soap, 0.33% of soda ash, and 2.6–2.7% of methyl cellulose. The soap and soda ash are dissolved in boiling water, and an aqueous 5% solution of methyl cellulose added. This is warmed and the melted wax added. The product is then stirred while cooling to give a good emulsion.<sup>3a</sup>

## FORMULA No. 3b

63 parts of a 5% aqueous solution of methyl cellulose are mixed into 1 part of potassium carbonate and 3 parts of soap

<sup>38</sup> Ibid., Vol. 1, p. 418 (1933).

<sup>&</sup>lt;sup>1</sup> P. C.

Bennett, Chem. Formulary, Vol. 4, p. 420 (1939).
 Ibid., Vol. 5, p. 533 (1939).

dissolved in 37 parts of boiling water. The mixture is cooled to 70°C. and 100 parts of a melted mixture containing 1 part of beeswax, 1 part of paraffin and 8 parts of ceresine added. Heating is continued for a half hour, the mixture is stirred well, the amount of water which has evaporated is replaced, and 96 parts of white spirit or turpentine substitute are gradually added. The whole is stirred until cool.<sup>3b</sup>

## FORMULA No. 4.

Beeswax Yellow	5 lb.	Turpentine	3 gal.
Paraffin Wax	4 lb.	Salts of Tartar	1½ lb.
Soap Chips	3 lb.	Water	3½ gal.
Stearic Acid	3 lb.		, -

Dissolve salts of tartar and soap in boiling water. Melt waxes in another container and heat to 200°F. when the boiling water soap solution is added slowly with vigorous stirring until homogeneous. Turn off heat and run turpentine in slowly with good stirring. Pack in cans when cold.<sup>4</sup>

#### FORMULA No. 5.

Carnauba Wax Beeswax Ceresin	6.0 4.0 4.0	Oleic Acid Triethanolamine Water	7.0 2.5 75.0
Turpentine	70.0	Petroleum <sup>5</sup>	40.0
Trichlorethylene	2.0		

#### FORMULA No. 6.

Carnauba Wax Beeswax	33 66	Soap Water	1 10
Turpentine or White			
Spirit	225		

The soap is dissolved in water (hot) and the waxes are dissolved in the dipentene. When cool the solutions are mixed with vigorous shaking or stirring.<sup>6</sup>

<sup>3</sup>b Ibid.

<sup>4</sup> Ibid., Vol. 1, p. 421 (1933).

<sup>&</sup>lt;sup>5</sup> Ibid., Vol. 4, p. 432 (1939).

<sup>6</sup> P. C.

#### FORMULA No. 7.

Carnauba Wax #2	10	Triethanolamine	4
Candelilla Wax	10	Borax	11/2
Oleic Acid	3	Water 7	185

#### FORMULA No. 8.

Carnauba Wax	60	oz.	Caustic Soap	11/2	oz.
Dry Castile Soap		oz.	Water to make	4	gal.
Oleic Acid		oz.			

Heat the wax, soap, and acid to 100°C. and while stirring rapidly add the caustic dissolved in the water, heated to 80°C., slowly.8

## BRIGHT DRYING (RUBLESS) FLOOR POLISH EMULSIONS

## FORMULA No. 1.

Carnauba Wax No. 2	120.0	Caustic Soda (47%)	3.5
Oleic Acid	13.7	Water	843.5
Trigamine	19.0	-	

Melt the wax in the oleic acid, add Trigamine and heat to 90°C. Then add the caustic soda and 25 cc. of water at about 95°C. Stir until jell is formed. Then add the rest of the water which must be boiling. Stir thoroughly till cool.<sup>1</sup>

## FORMULA No. 2.

Carnauba Wax	120	Caustic Soda (50%)	8
Oleic Acid	20	Water	32
Trigamine	20	Water <sup>2</sup>	800
	FORMULA	No. 3.	
Carnauba Wax	13.20	Water,	108.00
Oleic Acid	1.50	Shellac	2.20
Triethanolamine	2.10	Ammonia (28%) <sup>3</sup>	0.32

1.00

Borax

<sup>&</sup>lt;sup>7</sup> P. C.

<sup>8</sup> P. C.

<sup>&</sup>lt;sup>1</sup> Bennett, Chem. Formulary, Vol. 4, p. 419 (1939).

<sup>&</sup>lt;sup>2</sup> Bennett, Chem. Formulary, Vol. 4, p. 419 (1933).

<sup>3</sup> Ibid., Vol. 1, p. 420 (1933).

## FORMULA No. 4.

Carnauba Wax No. 1	10 g.	Water	90	CC.
Ammonium Linoleate		Turpentine	1	cc.
Paste	g	Pine Oil		cc.
Borax	2 g.			

Heat the wax, turpentine and oil to 100°C. in a jacketed vessel fitted with a high speed stirrer. To this add slowly the paste, borax, and water previously heated to 100°C. Stir vigorously until emulsified.<sup>4</sup>

## FORMULA No. 5.

Carnauba Wax No. 1	20 g.	Ammonium Hydroxide		cċ.
Ammonium Linoleate		Water	180	cc.
Paste		Ammonium Hydroxide		cc.

Heat the wax to 100°C. in a jacketed vessel fitted with a high speed stirrer and to it add slowly the paste, water and 7½ cubic centimeters of the ammonium hydroxide dissolved and heated to 80°C. As soon as emulsified add the balance of the ammonium hydroxide while stirring vigorously.<sup>5</sup>

## FORMULA No. 6.

Carnauba Wax	13.2	Shellac	2.0
Duponol G	2,0	Ammonia	0.3
Borax	1.0	Water	81.5

Melt together, in a steam jacketed vessel, the carnauba wax and Duponol G. While stirring efficiently, add the borax, dissolved in 1% of the water (boiling) and stir until homogeneous. After a homogeneous emulsion is formed turn off the steam and stir slowly until cooled to room temperature.

#### FORMULA NO. 7.

a. Carnauba Wax	11.2	b. Shellac	1.5
Oleic Acid	2.4	Morpholine	0.2
Morpholine	2.2	Water	15.5
Water	67.0		

<sup>&</sup>lt;sup>4</sup> Ibid., Vol. 2, p. 423 (1935).

<sup>5</sup> Ibid.

<sup>&</sup>lt;sup>6</sup> E. I. du Pont de Nemours Co., Wilmington, Del.

Melt the carnauba wax carefully with the oleic acid and maintain the temperature closely at 90°C. Stir until well mixed, add the morpholine, and stir constantly until the whole mass is quite clear. In the meantime, the water should have been brought to a simmer in a separate kettle. Add it slowly to the hot wax mixture with steady stirring, making certain that each small portion is well incorporated before further addition. The mixture becomes increasingly viscous and should be of the appearance of petrolatum when two-thirds of the water has been added. After this stage has been reached, the mixture begins to thin out and the remainder of the water may be added rapidly. The total time for adding the water should be 30–40 minutes. A steam-jacketed kettle and a hand-operated paddle, or slow-speed, large-bladed propeller are recommended for successful production.

Allow the mixture to cool. While slowly stirring, add the shellac solution. This has been made by warming together the morpholine, water and shellac indicated in b above and filtering if necessary.

FORMULA	No.	8.
TOKMOTH	T10.	v.

Carnauba Wax	64.8	Oleic Acid	15.3
Paraffin Wax		Boiling Water	430.0
Morpholine	14.4	Cold Water 8	100.0

#### FORMULA No. 9.

2 0.10 11	Triethanolamine Oleic Acid	<b>3</b> 5
2 0.10 11	30 Oleic Acid 10	

Melt the wax and the oleic acid in boiling water bath. When the mixture is homogeneous, add the triethanolamine and stir until it is all uniformly incorporated.

Heat the water to boiling and add it slowly, portion by portion, permitting each addition to be well agitated before

<sup>&</sup>lt;sup>7</sup> Bennett, Chem. Formulary, Vol. 4, p. 418 (1939).

<sup>8</sup> Bennett, Chem. Formulary, Vol. 4, p. 419 (1939).

the next addition. As the first additions of water are made, the mixture thickens considerably and becomes gel-like in consistency. As more water is added, the mixture becomes like a cream. When still more water is added, it breaks into an emulsion and then becomes a watery product. During the gel and cream stage, it is very important that the addition of water be made carefully and efficient agitation be used in order to obtain a thoroughly homogeneous mass. It is just as detrimental to add the water too slowly as too fast. Equally as important as efficient agitation and careful addition of

is the maintenance of high temperatures during the

## . Make a Manila resin solution as follows:

Manila DBB	30	Ammonium Hydroxide	
Water	158	(28-29%)	12

Powder the resin and agitate it with water and the ammonia until all the resin is dissolved. Strain the solution.

3. Prepare the finished polish as follows:

Carnauba Wax Emulsion 34 Manila DBB Solution 39

Mix the two well, using stirring.9

## FORMULA No. 10.

Paraffin Wax	15	Borax )	Previously	71/2
Oleie Acid	15	Water (	dissolved	71/2
Triethanalamine		,		

Warm together to 90°C, and mix with an electric mixer. While keeping at 90–100°C, and stirring vigorously add the following which must be at 90–95°C.

Wax 100 Water

Cool quickly and package.10

<sup>:</sup> Hid: Vel: 5, p. 532 (1941):

#### FORMULA No. 10a.

Carnauha Wax

30 Rosin

6

Heat above to 140°C., cool to 100°C. and add following with vigorous stirring which has been heated to 95–100°C.

Soap Flakes	10	Water 108	270
Turpentine	1		

#### FORMULA NO. 11.

Carnauba Wax No. 1 Yellow	30	Water	420
Thermally Processed Congo	30	Triethanolamine	4
Sodium Hydroxide	1/2	Oleic Acid	9

Melt the wax and add the Congo at about 125°-150°C. Hold until the mixture is homogeneous. Put the mixture in a boiling water bath. Add the oleic acid slowly and with vigorous stirring. Do the same with the triethanolamine. Dissolve the sodium hydroxide in 25 parts of water. Heat the solution to its boiling point and add it slowly to the mixture still in the boiling water bath, using vigorous stirring. When the mixture is thoroughly homogeneous, add the remainder of the water (also at its boiling point, and also with vigorous stirring).<sup>11</sup>

#### FORMULA No. 12a.

"Rilan" Wax	10	Soda	2
Japan Wax	10	Water 12a	100
Paraffin Wax	5	_	

#### FORMULA No. 12b.

Montan Wax, Bleached	8	Potash	1
Soft Soap	2	Water 12b	85
Rosin	1		

<sup>&</sup>lt;sup>108</sup> Bennett, Chem. Formulary, Vol. 1, p. 421 (1933).

<sup>&</sup>lt;sup>11</sup> Ibid., Vol. 5, p. 532 (1941).

<sup>&</sup>lt;sup>12a</sup> J. Davidsohn and A. Davidsohn, Shoe Creams & Polishing Waxes (1939).

<sup>12</sup>b Ibid.

#### FORMULA No. 12c.

Montan Wax, Bleached	5.6	Potash	1.5
Japan Wax	3.0	Water	55.0
Paraffin (50-52°C.)	4.0	Solvent 120	25-30
Curd Soap or Soft Soap	2.0		

#### FORMULA No. 12d.

35 parts spindle oil and 30 parts tall oil are mixed and warmed to about 70°C., then 8 parts of caustic soda solution, 38° Bé., is slowly added with constant stirring and boiled until saponification takes place. Then 25 parts of spindle oil, and 15 parts of kerosene are added as well as 5–10 parts of hot water.<sup>12d</sup>

	FORMULA No. 12e.
Olein	5 Alcohol
Resin	6

are melted together and mixed with good stirring at about 70°C: with 2.5 parts of caustic soda solution (38° Bé.). Then add and mix in turn.

Kerosene	70	Water 12e	5-10
Turkey Red Oil			

## FURNITURE POLISHES

#### FORMULA NO. 1.

Bleached Beeswax	2	Stearic or Oleic Acid	31,2
Carnauba Wax	6	Triethanolamine	154
Halowax No. 1013	4	Water	63
Turpentine	20	Perfume	To suit

Melt the three waxes and add the turpentine with agitation. Prepare the soap solution by heating the triethanolamine dissolved in the water almost to the boil and adding the molten stearic acid or liquid oleic. The soap may also be pre-

Ibid.

J. Davidsohn and A. Davidsohn, Since Cream & Polishing Waxes (1939).

12e Thid.

pared by heating the triethanolamine and water with acid in powdered form. Run the former solution into the latter with agitation. Continue to agitate until cool. If erly made the emulsion is

	FORMULA	No. 2.	
Blendene Spindle Oil Perilla Oil	20 40 20	Varnolene Water Borax <sup>2</sup>	40 120 2
	FORMULA	No. 3.	
Carnauba Wax Beeswax Ceresin Wax Turpentine	30 15 15 26	Naphtha Stearic Acid Triethanolamine Water <sup>3</sup>	24 8 4 65
	Formula	No. 4.	
Carnauba Wax Beeswax Ceresin Wax Naphtha	10.0 4.0 4.0 80.0	Stearic Acid Triethanolamine Water <sup>4</sup>	8.0 4.5 200.0
	FORMULA	No. 5.	
Carnauba Wax Bleached Japan Wax Paraffin Wax Turpentine	6 3½ 1½ 12	White Curd Soap Rosin, Pale Water Clove Oil <sup>5</sup>	3 2 30 Trace
	FORMULA	No. 6.	
Carnauba Wax Paraffin Wax Ceresin Naphtha Turpentine	6.0 9.0 2.0 43.0 4.0	Trihydroxyethylamine	1.0 4.5 130.0
	FORMULA	No. 7.	
A. Carnauba Wax Turpentine Stearic Acid	60 60 2	B. Trihydroxethylamine Stearate Water	12 62

<sup>&</sup>lt;sup>1</sup> Bennett, Chem. Formulary, Vol. 4, p. 432 (1939).

<sup>&</sup>lt;sup>2</sup> Ibid., Vol. 5, p. 534 (1941).

<sup>&</sup>lt;sup>3</sup> Ibid., Vol. 1, p. 422 (1933).

<sup>4</sup> Ibid.

<sup>5</sup> Ibid.

<sup>&</sup>lt;sup>6</sup> Bennett, Chem. Formulary, Vol. 1, p. 423 (1933).

## ish Emulsions

Heat (A) and (B) in separate vessels to 200°F, and fun (B) into (A) slowly with vigorous stirring. Stop when homogeneous.7

## FORMULA No. 8.

Diglycol Stearate	10 oz.	Water	4 gal.
Paraffin Oil	3 pt.	Lemon Odor ("Lemenone")	2 oz.

The oil and diglycol stearate are heated together to 150°F. until the wax has melted. The hot wax mixture is then run into the water which has previously been heated to about 200°F. while stirring rapidly. Stirring should be continued tifitil the batch has cooled. The lemon odor is added after the batch has partially cooled.8

## FORMULA NO. 9.

Heat until wax is dissolved.9

#### FORMULA NO. 10.

Mineral Oil Kerosene		Triethylamine Wood Alcohol	2 2	oz. fl. oz.
	4½ fl. oz.	Water	60	fl. oz.
Stearic Acid	41/2 oz:			

Mix the mineral oil, turpentine and kerosene, to the stearle acid and heat to about 148°F, to melt and dissolve the stearic acid. This should produce a clear solution.

In another container the triethylamine, alcohol and water are mixed and heated to 148°F. This mixture is then added to the first hot solution and stirred vigorously for 15 minutes to produce a smooth creamy and white emulsion. The stirring should be brisk. Care should be exercised in handling tur-

<sup>₹</sup> Ibid.

<sup>8</sup> Crowley, Money Making Formulas: 9 Bennett, Chem. Formulary, Vol. 1, p. 422 (1933). 16 Crowley, Money Making Formulas.

pentine and kerosene because of their inflammability. Gentle stirring should be continued until the product is cool.

## FORMULA No. 11.

Naphtha	10	Nelgin
Thin Mineral Oil	72	Water
Perilla Oil	30	

Soak Nelgin in water for a few hours. Stir until dispersed. To this add oil mixture slowly with rapid stirring.<sup>11</sup>

## FORMULA No. 12.

Oleic Acid	2	2. Water	13⁄4
Kerosene	4	Sodium Hydroxide	1/20
Sulfonated Castor Oil	2	Triethanolamine	1/10

Mix 1 thoroughly. Mix 2 and pour into 1 while stirring at high speed. Continue to stir until batch is of uniform creamy consistency.<sup>12</sup>

## FORMULA No. 13.

Paraffin Oil	20	Gum Arabic	2
Red Oil	5	Water	70
Soft Soap	3		, ,

The above are mixed vigorously until completely emulsified.<sup>13</sup>

#### FORMULA No. 14.

Raw Linseed Oil	 Xylol	5
Spindle Oil	Soft Soap	1
Stoddard Solvent	Water 14	10
	 TT WELL	19

#### FORMULA No. 15.

Spindle Oil	40 g.	Varnolene	120 cc.
Perilla Oil	20 g.	Water	
Trigamine Stearate Denatured Alcohol	30 g. 10 cc.	Borax Lemenone 15	120 cc. 2 g. 5 cc.

<sup>&</sup>lt;sup>11</sup> Bennett, Chem. Formulary, Vol. 2, p. 421 (1935).

<sup>12</sup> Crowley, Money Making Formulas.

<sup>13</sup> Bennett, Chem. Formulary, Vol. 3, p. 287 (1936).

<sup>14</sup> Ibiá.

<sup>15</sup> Ibid., Vol. 5, p. 534 (1941).

#### FORMULA No. 16.

Turpentine	8 gal.	Antimony Chloride	4 gal.
Naphtha	30 gal.	Gum Arabic	10 lb.
Lt. Spindle Oil	49 gal.	Gum Tragacanth	10 lb.
Acetic Acid (36%)	6 gal.	Perfume	1 gal.
Water	100 gal.		J

Make up with water to 200 gallons and run through colloid mill.<sup>16</sup>

#### FORMULA No. 17.

Yellow Ceresine	3 lb.	Paraffin Oil (28°)	1 gal.
Japan Wax	1 lb.	Water	7 gal.
Beeswax	2 lb.	Carbonate of Potash	3 oz.
Linseed Oil, Raw	4 gal.	Soap Chips (Animal Fat	
Turpentine	1 gal.	Soap)	1 lb.

Mix the above thoroughly.17

#### FORMULA No. 18.

Water	10	Acetic Acid	1/8
Nut Oil	1	Gum Arabic 18	11
Mineral Oil	1		

#### FORMULA No. 19.

White Mineral Oil	2	Water	4
Kerosene	4	Perfume Oil	To suit
Sulfonated Castor Oil	1		

The kerosene and mineral oil are mixed and the sulfonated castor oil is added, while mixing vigorously. Next run in the water while mixing briskly. Finally add the perfume in the proportions of about ½ ounce to each gallon of polish.<sup>19</sup>

## FORMULA No. 20.

a. Montan Wax, Double		b. Soft (Potash) Soap	1
Bleached	4	Potassium Carbonate	3
Beeswax	5	Water	82
		c. Ammonia (0.910)	5

<sup>&</sup>lt;sup>16</sup> Ibid., Vol. 1, p. 422 (1933).

<sup>17</sup> Ibid.

<sup>18</sup> Bennett, Chem. Formulary, Vol. 1, p. 423 (1933).

<sup>19</sup> Crowley, Money Making Formulas.

Melt a, boil up with b, and stir until a smooth emulsion has formed. Cool. Add  $e^{2\theta}$ 

Eansers .	AT.	31
FORMULA	IN O.	21.

Crude Montan Wax	5	Potassium Carbonate	3
Beeswax	4	Water <sup>21</sup>	87
Seft Soap	į	Water.	

Boil all ingredients together until a smooth emulsion has formed. Stir cold.

## LEATHER POLISHES

## FORMULA NO. 1.

Carnauba Wax Montan Wax, Bleached Beeswax Japan Wax	5.0 5.0 2.0 1.0	Paraffin Wax Potash Water <sup>1</sup>	12.0 1.7 72.0
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## FORMULA No. 2.

Montan Wax, Bleached	10	Paraffin (50-52°C.)	13
Rosin	2	Potash	21:
Japan Wax Diglycol Stearate	1	Water 2	21 <sub>1</sub> 70

## FORMULA No. 3.

Carnauba Wax	12	Water	17=20
Stearin	31/2	Turpentine 3	12=15
Triethanolamine	1		- 1

## FORMULA No. 4.

C 1 ****			_
Carnauba Wax	11	Triethanolamine	1
Turpentine	16	Water	<b>9</b> 6
Stearic Acid	3	Water Sol. Nigrosine #	1
Oil Soluble Nigrosine	2		

<sup>20</sup> Bennett, Chem. Formulary, Vol. 5, p. 533 (1941).

<sup>21</sup> Ibid., p. 534.

<sup>1</sup> Davidsohn & Davidsohn, Shoe Creams & Polishing Waxes. 2 Ibid.

<sup>8</sup> Ibid.

<sup>\*</sup> Bennett, Chem. Formulary, Vol. 1, p. 424 (1933).

## FORMULA NO. 5.

Water Petassium Oleate	20 gal. 7½ lb. ½ lb.	Carnauba Wax Turpentine	6 lb: 4½ gal:
Trisodium Phosphate Beeswax, Yellow	1/2 lb: 6 lb:	Pine Oil Terpineol 5	4½ gal: ½ gal: ¼ gal:

## FORMULA No. 5a.

Mineral Spirits Light Mineral Oil Giyeeryl Monoricinoleate Ricinoleic Acid Water	169.00 187.50 93.60 11.03 624.75	Water solution of potassium hydroxide containing 1.65 parts of potassium hy= droxide	19,25
--	--	---	-------

The potassium hydroxide solution and one-third of the total water are put in a mixing tank. The risinoleic acid is then added and after the required stirring soap is formed by virtue of the action between the alkali and the fatty acid, the glyceryl mono ricinoleate is added followed by the mineral oil and in turn by one-half of the remaining water. Mineral spirits are added and finally the remaining water. The entire mixture after thorough agitation is then sent through an homogenizer to produce a commercial dispersion.58

## FORMULA NO. 6.

Beeswax Ceresin Wax Carnauba Wax Turpentine	1 lb: 1 lb: 6 0z: 3 pt:	Yellew Seap Oil Seluble Black Water	6 92: Enough to color Sufficient
--	----------------------------------	---	--

Shave the soap and dissolve in the smallest possible quantity of water by means of heat, melt the waxes together, add the turpentine and stir well, then add the anilin dye and stir in the soap solution, continuing to stir until cold.8

<sup>5</sup> Ihid., Vol. 2, p. 290 (1935).
52 U. S. 2,275,596.
6 Bennett, Chem. Formulary, Vol. 1, p. 430 (1933).

## FORMULA No. 7.

Beeswax	100	b. Water	200
Paraffin Wax	20	Potassium Carbonate,	
Carnauba Wax, Gray	8	Calcined	6
Naphtha	80	Glue 7	35
Turpentine	70		

## FORMULA No. 8.

#### WAX MASS

Bleached Carnauba Wax	10.0 kg.	Pale Rosin	0.5 kg.
Japan Wax	5.0 kg.		3.0 kg.
Bleached Beeswax	2.0 kg.	Stearin	0.5 kg.

## SAPONIFYING SOLUTION

White Tallow Curd Soap	0.75 kg.	Water	35.00 kg.
Potassium Carbonate	1.50 kg.	Turpentine	1.00 kg.
Caustic Soda (36° Bé.)	0.25 kg.		

# DYE SOLUTION FOR DARK REDDISH-BROWN CREAM (per 100 kg. of above mass)

Brown R	350 g.	Tartrazine X	50 g.
Mars Red Nigrosin G	250 g. 15 g.	Final additional water	40 kg.8

## FORMULA No. 9.

Carnauba Wax	6	Lemongrass Oil	1/2
Ceresin Wax	3	Light Mineral Oil	1
Candelilla Wax	3	Diglycol Stearate	5
Turpentine	5		

Heat together below 90°C. and stir till clear. Pour slowly while stirring with high speed stirrer into

Water (Boiling)

100

Stir until temperature falls to 70°C.9

## FORMULA No. 10.

Carnauba Wax	25 g.	Triethanolamine	1 cc.
Ceresin	10 g.		

<sup>&</sup>lt;sup>7</sup> Ibid., Vol. 4, p. 430 (1939).

<sup>8</sup> Ibid., p. 431.

<sup>&</sup>lt;sup>9</sup> Bennett, Chem. Formulary, Vol. 4, p. 430 (1939).

Melt together and stir; add following slowly:

Turpentine	30 cc.	Gasoline	30 cc.
Deodorized Kerosene	5 cc.		

For black polish add 1% oil black dye.

For brown polish add brown iron oxide sufficient to color. The above mix is poured into cans at 25°C. and allowed

The above mix is poured into cans at 25°C. and allowed to set.<sup>10</sup>

# FORMULA No. 11. Carnauba Wax 7.5 Potassium Carbonate 1.5 Marseilles Soap 1.0 Water 79.0

Melt the carnauba wax, and add the heated mixture of the other ingredients. Stir rapidly, and add 11 kg. nigrosine previously dissolved in a small amount of the soap solution.<sup>11</sup>

	For	MULA	No. 11a.		
Carnauba Wax Paraffin Wax Ozokerite V.M.&P. Naphtha Turpentine	30 45 10 215 20	g. g. g. cc.	Stearic Acid Triethanolamine Water Oil Soluble Black Water Soluble Black <sup>1</sup>	20 7½ 650 15 5	g. g. cc. g.
	For	MULA	No. 11b.		
Carnauba Wax Ceresin Wax Candelilla Wax		6 3 3	Lemongrass Oil Light Mineral Oil Diglycol Stearate		1/2 1 5

Heat together below 90°C. and stir until clear. Pour slowly while stirring with high speed stirrer into

Water (Boiling) 100

Turpentine

Stir until temperature falls to 70°C. The finished product is snow white, applies easily and rubs to a high gloss. If

<sup>&</sup>lt;sup>10</sup> Ibid., Vol. 2, p. 290 (1935).

<sup>&</sup>lt;sup>11</sup> Ibid., Vol. 1, p. 429 (1933).

<sup>&</sup>lt;sup>11a</sup> Bennett, Chem. Formulary, Vol. 4, p. 430 (1939).

a paste polish is desired reduce the water to about 50 to 60 parts. If a colored paste is desired an aniline dye is dissolved in th

	FokMula	No. 11ĉ:	
Pale Carnauba Wax Japan Wax Bleached Montan Wax Rosin Curd Soap	4.5	Potash Caustic Soda Solution Water Turpentine Water ite	1.5 0.2 40.0 2.0 30.0
	FORMULA	No. 11d.	
Bleached Montan Wax Carnauba Residues Japan Wax	14 7 3	Potash Water Türpentine iid	2½ 75 2
	Formula	No. 11e.	
Pure Carnauba Wax Japan Wax Carnauba Wax Residues Parattin Wax	2.0 5.0 13.0 1.0	Potash Water	9.7 2.5 40.0 25.0
	FORMULA	No. 11 f.	
Montan Wax Beeswax Carnauba Wax Paraffin Wax (50=52°C:) Rosin		Potash Water Curd Seap Water itt	15 300 2 70
	FORMULA	No. 11g.	
Mentan Wax IG Wax O: P: IG Wax E: Japan Wax Rosin		Paraffin Wax (50-52°C.) Potash Water Water 11g	1.5 1.5 45.0 40.0

<sup>116</sup> fisid. 116 Davidsohn & Davidsohn, Shoe Creams & Polishing Waxes: 11d Davidsohn & Davidsohn, Shoe Creams & Polishing Waxes:

iie Ibid

<sup>118</sup> Davidsohn & Davidsohn, Shee Creams & Polishing Waxes.

ļ	Polish B	mulsions	381
	FORMULA	No. 11h.	
"Rilan" Wax 1 S Beeswax Paraffin Wax (50-52°C.)	8.0 5.0 7.0 3.0 7.0	Rosin Curd Soar Potash Water Water 118	0.5 0.5 0.5 50.0 30.0
	FORMULA	No. 11i.	
Carnauba Wax Paraffin Wax (50-52°C.) Ro Diglycol Stearate	12,50 1,00 2,50 1,50	Borax Potash Water Water <sup>1</sup>	0.75 2.50 50.00 25.00
	FORMULA	No. 11j.	
Carnauba Wax Stearin	85 9	Triethanolamine Water	4 400

The stearin is melted down and the

in hot water, is added at about 70°C. and then the whole stirred and heated to 100°C. Into this solution the melted carnauba wax, heated to 80–90°C. is stirred in, and the stirring continued until the whole mass has cooled.<sup>113</sup>

## FORMULA No. 11k.

Montan Wax, Bleached	20	Borax	
Japan Wax	7	Curd Soap	2
Carnauba Wax Residues	3	Water	40
Paraffin Wax (50-52°C.)	5	Turpentine or White	
Rosin	2	Spirit 11k	20
Potash	2		

#### FORMULA

Wax, Bleached	7.5	Curd Soap	1.5
Carnauba Wax Residues	12.0	Potash	1.0
Japan Wax	6.0	Water	40.0
Paraffin Wax	5.0	Water	5.0
Rosin	1.0	Ammonia (0.910)	0.5

0.5 parts of ammonia and 5 parts of water are only added after addition of the alkali lye and after putting in the emul-

<sup>11</sup>h Thid

<sup>111</sup> John, White Shoe Dressings and Cleaners (1940).

<sup>111</sup> John, White Shoe Dressings & Cleaners (1940).

<sup>1</sup>k Ibid

sifier. 20 parts of turpentine or of a solvent composition (as for example 10 parts turpentine and 10 parts white spirit) may be added.111

#### FORMULA No. 11m.

Montan Wax, Bleached	6	Rosin	1/2
Carnauba Wax	31/2	Curd Soap	1/2
Beeswax	3	Potash	3/4
Japan Wax	2	Water	50
Paraffin Wax (50-52°C.)	4	Turpentine	28

#### FORMULA NO. 12.

#### Mixture 1

Carnauba Wax Montan Wax	8	Paraffin Wax	4

These are saponified in a hot solution of

Potash 3 W	Vater 50
------------	----------

Replace any evaporation with additional warm water.

#### Mixture 2

No. 1 Polish Black 4 Wat	er 25
--------------------------	-------

These should be milled together in a color mill until thoroughly dispersed.

While Mixture No. 1 is hot, add Mixture No. 2 slowly and with constant stirring. As it cools, the mass will slowly set to a paste. Before it is too stiff for flowing pour into suitable containers and set aside until cold.12

#### FORMULA No. 13.

Carnauba Wax	12.0	Sodium Nitrate	0.1
Rosin	0.5	Water 13	To make 100.0
Triethanolamine Oleate	3.5		

<sup>111</sup> Ibid.

<sup>&</sup>lt;sup>11m</sup> John, White Shoe Dressings & Cleaners (1940).

<sup>12</sup> Bennett, Chem. Formulary, Vol. 1, p. 430 (1933).

<sup>13</sup> P. C.

P	olish E1	nulsions	383
	Formula	No. 14.	
	WAX N	MASS	
Crude Montan Wax Beeswax Carnauba Wax	10 4 1	Japan Wax Shellac Wax	5 1 <u>4</u> 4
SAPO	NIFYING	SOLUTION	
Potassium Carbonate Boiling Water Borax	1.5 75.0 0.5	Diglycol Stearate Palm Oil Soap	1.0 1.5
· I	OYE SOL	LUTION	
Water Nigrosin	50 5	Final additional water 150 k	rg.14
	Formula	No. 15.	
Montan Wax Crude Japan Wax Paraffin Wax (50°-52°C.) Carnauba Wax Residue Carnauba Wax	4.00 0.30 1.00 2.00 0.50	Water	0.70 0.30 1.15 30.00 10.00
	FORMULA	No. 16.	
Montan Wax, Crude Montan Wax, Double Bleached Rosin Paraffin Wax (50/52°C.) Potassium Carbonate	3.00 2.00 2.00 2.20 2.20	Soap, Castile Sodium Tetraborate Nigrosine, Water Soluble Metanil Yellow Dye Water	0.30 0.30 2.00 0.08 95.00
Pour at 65°C.16			
	FORMULA	No. 17.	
Montan Wax, Crude Paraffin Wax (40/42°C.) Rosin Potassium Carbonate Pour at 40–45°C. <sup>17</sup>	15 20 5 3	Water Metanil Yellow Yellow Mandarin Dye, Water Soluble	96 0.9 0.8
<ol> <li>Ibid., Vol. 4, p. 431 (1</li> <li>Bennett, Chem. Formu</li> <li>Ibid., p. 432.</li> <li>Ibid.</li> </ol>		4, p. 431 (1939).	

Carnauba Wax

## Practical Emulsions

Montan Wax, Crude Paraffin Wax, Soft Rosin Potassium Carbonate	Formula 15 20 4-6 3	No. 18. Water Oil Soluble   Mandarin Yello Color   Metanil Yellow	95 9 <b>W</b> 1 ; 18 1
	Formula	No. 19:	
	WAX :	MAŠŠ	
Crude Montan Wax Rosin	20.0 5.0		10.0
SAPO	ONIFYIN	solution	
Potassium Carbonate	6.0	Water	$\hat{6}\hat{0}.\hat{0}$
	DYĒ ŠOI	Lution	
Nigresin T. Water	5.5 50.0	Final additional water 18	50.0
	Formula	No. 20.	
Montan Wax (Bleached) Montan Wax (Crude) Color L34 Candelilla Wax	10 5 6 4	Paraffin Wax Diglycol Stearate Soap (Powdered) Water	5 3 2 50

Melt the waxes, color and diglycol stearate together. Dis solve the soap in the water which must be boiling and add slowly with stirring to the melted waxes. When the temperature has fallen to 80°C, add the turpentine slowly with thorough agitation. Pour at 55°C.

Turpine

For colors, use oil soluble colors dissolved in the waxes. 86

## FORMULA No. 21.

1. Trihydroxyethylamine		Carnauba Wax	40
Stearate	25	Turbentine	20
Beeswax	10	2. Water 21	500
Candellila Wax	30		200

<sup>18</sup> Ibid., Vol. 5, p. 536 (1941). 18 Ibid., Vol. 4, p. 431 (1939). 28 Dennett, Chem: Formulary, Vol. 4, p. 429 (1939): 21 Ibid., Vol. 1, p. 428 (1933):

#### FORMULA No. 22.

Shoe Stain:		b. Shellac	20.0
a. Montain Wax Crude	15.0	Sodium Tetraborate	7.0
Rosin	2.0	Water	75.0
Paraffin Wax (40/42°C.)	3.0	c. Nigrosin, Water Soluble	5.5
Potassium Carbonate	0.5	Water	25.0
Soap, Castile	4.0		
Water	65.0		

a, b, c, are prepared separately and then mixed together.22

## FORMULA No. 23.

a. Vandyke Brown	8 1ь.	Tragacanth Solution	1/2
Raw Umber	30 lb.	b. Water	gal.
Casein Solution	4½ gal.	Caustic Soda	1b.
Water	1¼ gal.	Castile Soap	2 lb.
	,,,	Gray Carnauba Wax	6 lb.
		Black Montan Wax	3 lb.
		Nigrosine	6 oz.
		Rogers Fish Glue 23	1½ gal.

Mix solutions of a and b.

## FORMULA No. 24.

a.	Burnt Turkey Umber	20	1b.	b. Water	63/4 gal.
	Indian Red	3	1b.	Castile Soap	1 lb.
	Middle Chrome	2	lb.	Gray Carnauba Wax	5¼ 1b.
	Casein Solution		gal.	Water Brown Dyestuff	$1\frac{1}{2}$ oz.
	Garnet Shellac			-	-
	Solution		gal.		

These two finishes are made in sections as above, the pigment paste being added to the wax solution either hot or cold. Usually no dyestuff is necessary, but for these darker shades, a trace of nigrosine or dark brown color is advisable. Both finishes are full bodied and will stand considerable dilution with either cold water or casein solution.<sup>24</sup>

<sup>&</sup>lt;sup>22</sup> Ibid., Vol. 4, p. 432.

<sup>&</sup>lt;sup>23</sup> Davidsohn & Davidsohn, Shoe Creams & Polishing Waxes (1939).
<sup>24</sup> Ibid.

## FORMULA No. 25.

Water	1 gal.	Carnauba Wax	$2\frac{1}{2}$ lb.
Nigrosine	3 oz.	Nigrosine Stearate	1/4 lb.
Ethanolamine	¼ lb.	Turpentine	
Stearic Acid	3/4 lb.	Clove Oil	

The wax is melted in one container and diluted with the solvents and then vigorously stirred into the second receptacle with the boiling soap and alkaline solution.<sup>25</sup>

#### FORMULA No. 26.

a. Crude Montan Wax	18	b. Water	260
Japan Wax	2	Potash (98%)	6
Carnauba Wax	4	Water-Soluble Nigrosin	12
Rosin			

Heat A and B separately to 95–100°C, and add B to A while stirring vigorously with an electric mixer.<sup>26</sup>

## FORMULA No. 27.

## BLACK BOTTOM GLOSS

Water	1 gal.	Japan Wax	2 lb.
Potassium Oleate	6 oz.	Turpentine	½ gal
Nigrosine	3∕4 lb.	White Spirit	½ gal
Paraffin Wax	2 lb.	Cassia Oil	5 oz.
Black Montan Wax	31/2 lb,		

These brown and black glosses are prepared in the same way as the white gloss: the waxes and solvents being mixed separately and added to the tinted boiling water.<sup>27</sup>

#### FORMULA NO. 28.

## BROWN BOTTOM GLOSS

Water	1 gal.	Turpentine	2 gal
Castile Soap	3½ oz.	Terpineo1	½ pt.
Potassium Carbonate	1½ oz.	No. 62995 Water Brown	
White Montan Wax	3 lb.	Dvestuff 28	1½ oz.
Yellow Beeswax	3 1b.	•	. /-

<sup>25</sup> Ibid.

<sup>&</sup>lt;sup>26</sup> Bennett, Chem. Formulary, Vol. 1, p. 430 (1930).

<sup>&</sup>lt;sup>27</sup> J. Davidsohn & A. Davidsohn, Shoe Creams & Polishing Waxes (1939).

<sup>28</sup> Thid.

## FORMULA No. 29.

## "NATURAL" BOTTOM FINISH

a. Titanox or Lithopone	14	1b.	b. Water	16	gal.
China Clay	13	lb.	Soap	$3\frac{1}{2}$	lb.
Yellow Ochre	10	lb.	Gum Arabic	6	1b.
Casein Solution	4	gal.	Yellow Carnauba Wa	ax 103/4	lb.
Bleached Shellac			Fish Glue 29	7	pt.
Solution	2	gal.			

## FORMULA No. 30.

## PALE PINK BOTTOM FINISH

a. Pale Buff (No. 8102			b. Water	11/2	gal.
Pigment)	6	1b.	Gum Tragacanth	1	qt.
Bleached Shellac			Potassium Oleate	1/4	1b.
Solution	1	gal.	Yellow Carnauba Wax	13/4	1b.
Water	3	pt.	Fish Glue 30	3	pt.

## FORMULA No. 31.

## SHOE INK

Water	12 gal.	Carnauba Wax	6	lb.
Logwood Extract	1¼ gal.	Beeswax	2	1b.
Gum Arabic	3∕4 lb.	Ferrous Sulphate 31	11/4	1b.
Soft Soap	1¼ lb.			

## FORMULA No. 32.

## MODERN TRANSPARENT HEEL AND EDGE FINISH

Water	1½ gal.	Gum Tragacanth	3∕₄ oz.
Borax	1 oz.	Castile Soap	3 oz.
T.N. Shellac	4 oz.	Gray Carnauba Wax	3∕ <sub>4</sub> 1b.
Brown Water Dyestuff	6½ oz.		

The ingredients are boiled together until solution is complete and to prevent any separation of the wax, the whole is cooled down as quickly as possible with constant stirring.<sup>32</sup>

<sup>29</sup> Ibid.

<sup>30</sup> Davidsohn & Davidsohn, Shoe Creams & Polishing Waxes (1939).

<sup>31</sup> Ibid.

<sup>32</sup> Ibid.

## FORMULA No. 33.

## DARK BROWN HEEL AND EDGE STAIN

## SOLUTION No. 1

Water Borax Ammonia (0.880)	,, ,	Dark Brown Dyestuff Lactic Casein	12 oz. 34 lb.
	Solution	N No. 2	
Water Sodium Stearate Algin		Shellac Wax Nigrosine	1½ lb. 1 oz.

Solution No. 1 is prepared and allowed to become quite cold. Solution No. 2 is boiled until all the wax has emulsified and the cold dark brown dye solution is stirred into it rapidly. This prevents the separation of any wax when the finish is cold.33

## FORMULA No. 34.

## "NATURAL LEATHER" BOTTOM STAIN

Prepare three solutions as follows and allow the first two to become cold:

a.	Gum Tragacanth			c. Castile Soap	31/2	oz.
	(Powdered)	2	oz.	Yellow Carnauba Wax	17	oz.
	Methylated Spirits	4	oz.	Water	1	gal
	Water	1	gal.	Primrose Yellow		
ь.	Garnet Shellac	2	lb.	Dyestuff	1/2	oz.
	Borax	1/2	1b.			
	Water	1	gal.			

When solution c is boiling rapidly stir in 1 quart each of the tragacanth and shellac solutions, and cool the whole off rapidly.34

<sup>33</sup> Davidsohn & Davidsohn, Shoe Creams & Polishing Waxes. 84 Ibid.

#### FORMULA No. 35.

## BROWN BOTTOM FINISH

a. Raw Umber	35	1b.	b. Soft Water	10 gal.
Chrome Yellow	5		Tragacanth Solution	1 gal.
Garnet Shellac Solution	n 4		Sodium Stearate	1¼ lb.
Water	2		Gray Carnauba Wax	7¼ lb.
vv ater	4	gai.	Liquid Fish Glue 85	$\frac{7}{4}$ ID. $\frac{3}{4}$ gal.

## FORMULA No. 36.

## LIGHT BROWN RUSSET FINISH

b. b. Water 3 gal.
b. Ammonium Linoleate 3/4 lb.
b. Gray Carnauba Wax 36 234 lb.
•
gal.
al.
al.

## FORMULA No. 37.

## "BEND" COLOURED BOTTOM FINISH

a. Yellow Ochre	6	lb.	b. Water	7 gal
Lemon Chrome	3	lb.	Shellac Solution	2½ gal
Titanox or Kaolin	16	lb.	Castile Soap	1½ lb.
Water	2	gal.	Carnauba Wax	6¼ lb.
		•	Fish Glue 37	5½ pt.

## FORMULA No. 38.

## WAX RUSSET FINISH (BUFF)

a.	Soft Water	8	gal.	b. Soft Water	5	gal
	Castile Soap	3	lb.	China Clay	40	1b.
	Fish Glue	10	1b.	Yellow Ochre	10	1b.
	Yellow Carnauba	Wax 6	1b.	Red Oxide	3	1b.
	Japan Wax	2	1b.	Citronella Oil 38	11/2	lb.

<sup>35</sup> Davidsohn & Davidsohn, Shoe Creams & Polishing Waxes.

<sup>87</sup> Ibid.

<sup>38</sup> Davidsohn & Davidsohn, Shoe Creams & Polishing Waxes.

## FORMULA NO. 39. BROWN RUSSET STAIN

Soft Water	3¼ gal.	Borax	½ lb.
Farina	5 oz.	Orange Shellac	2 lb.
Castile Soap	⅓ lb.	Burnt Turkey Umber	15 lb.
Fish Glue	3½ pt.	Boiled Linseed Oil	2½ pt.
Yellow Carnauba Wax	1¼ lb.	Sassafras Oil	½ pt.

This finish is prepared in two parts, the water being divided up as above, and the wax emulsion added to the colour paste: the whole is then passed once or twice through a paint grinding mill.<sup>39</sup>

Formula No. 40.
"NATURAL LEATHER" FINISH

Soft Water	7	gal.	Fish Glue	3	qt.
Irish Moss	1∕4	1b.	China Clay	34	1b.
Castile Soap	1/2	1b.	Flake White	8	1b.
Yellow Carnauba Wax	21/4	1b.	Middle Chrome	31/2	lb.
Borax	1/2	1Ь.	Citronella Oil	11/2	pt.
T.N. Shellac	2	1b.			_

The wax and glue solution is prepared by boiling all the ingredients together, and then cooling down as quickly as possible so that there is no separation of the waxes. The earth pigments are ground up into a stiff paste with the water and the citronella oil, and the wax emulsion added to it: the whole is then passed through a paint mill.<sup>40</sup>

# FORMULA No. 41. RED-BROWN HEEL AND EDGE FINISH

Make two solutions of shellac and casein containing the equivalent of 2 lb. and 1 lb. of each per gal., using borax and ammonia as described in the preceding formulae. Mix one pint of each together to form a stiff jelly.

<sup>39</sup> Thid.

<sup>40</sup> Ibid.

## Prepare a colored wax solution as follows:

Water	3½ pt.	Brown Dyestuff	31/2	οz.
Castile Soap	$2\frac{1}{2}$ oz.	Gray Carnauba Wax	10 c	oz.
Caustic Soda	½ oz.			

and when boiling stir in the cold casein-shellac jelly; cool down as rapidly as possible.41

## FORMULA No. 42.

## WHITE BOTTOM GLOSS

Water	1	gal.	Carnauba Wax	8	1b.
Trihydroxyethlamine		_	Stearic Acid	₹4	1Ь.
Stearate	2	lb.	Turpentine	11/2	gal

This gloss can be prepared in the same way as the preceding one or alternatively in two pans; the wax, stearic acid and turpentine are melted and mixed together and then rapidly stirred into the boiling water and trihydroxyethylamine stearate.<sup>42</sup>

#### FORMULA No. 43.

## WHITE BOTTOM CREAM

Water Potassium Stearate	3 ¾	gal. 1b.	Dekalin Turpentine	2½ 2½	gal gal
Beeswax	6	1b.	Clove Oil 43	5	oz.
Yellow Carnauba Wax	6	1b.			

## FORMULA No. 44.

#### BROWN BOTTOM CREAM

Water	3	gal.	Turpentine	5	gal
Pearl Ash	1/2	1b.	Lavender Oil	1/2	1b.
Potassium Stearate	1/4	1b.	Wax Brown Dyestuff	1/2	oz.
White Beeswax	9	1ь.	Orange II.44	₹/8	oz.
Yellow Carnauba Wax	3	1b.			

Davidsohn & Davidsohn, Shoe Creams & Polishing Waxes.

<sup>48</sup> Ibid.

<sup>44</sup> Davidsohn & Davidsohn, Shoe Creams & Polishing Waxes.

#### FORMULA No. 45.

## ONE SETTING EDGE INK

	inoleate 1¾ lb. a Wax 8 lb. 2 lb. 10 gal. 1¼ lb.	(1½ lb. per ( Ammonia	
--	--	--------------------------	--

The shellac-nigrosine solution is prepared by boiling and is allowed to become quite cold. The wax emulsion is boiled for half-an-hour and the dye solution added rapidly with vigorous stirring; the casein is then poured in slowly and the agitation continued until the ink is quite

#### FORMULA No. 46.

# COMBINED INK FOR BOTTOMS, HEELS AND EDGES

Soft Water	35	gal.	Caustic Soda	3	1b.
Irish Moss	21/2	oz.	Gray Carnauba Wax	16	1b.
Borax	2	1b.	Fibre Wax	81/2	1b.
Garnet Shellac	8	1b.	Nigrosine	42	1b.
Castile Soap	3	1b.			

The water is brought to the boil and the ingredients are added in the order indicated. (Care must be taken with the caustic soda, to add only small amounts at a time.) When the whole is completely in solution, the heat is taken away and the ink cooled down quickly by running cold water through the jacketed pan, mechanical stirring being maintained the whole time. 46

<sup>45</sup> Ibid.

<sup>46</sup> Davidsohn & Davidsohn, Shoe Creams & Polishing Waxes.

## FORMULA No. 47.

#### GENERAL INK

Soft Water	35 gal.	Black Montan Wax	12	lb.
Irish Moss	$5\frac{1}{2}$ oz.	Gray Carnauba Wax	4	1b.
Borax	1⅓ lb.	Fibre Wax	8	lb.
Garnet Shallac	6 lb.	Nigrosine	38	1b.
Castile Soap	$3\frac{1}{2}$ lb.	Metanil Yellow	2	1b.
Caustic Soda	23/4 lb.			

#### FORMULA No. 48.

## ONE SETTING EDGE INK

Water	5	gal.	Carnauba Wax	3/4	1b.
Borax	5	oz.	Nigrosine	43/4	1b.
Bleached Shellac	11/4	lb.	Metanil Yellow	4	oz.
Caustic Soda	1/2	lb.	Ammonia	5.	oz.
Black Montan Wax	23/4	1b.			

## FORMULA No. 49.

## POLISHING INK

a. Water	80.0	b. Water	20.0
Marseilles Soap	3.0	Methyl Cellulose	1.0
Carnauba Wax	4.0	c. Turpentine Oil	1.0
Japan Wax	2.0	Citronella Oil	0.3

Boil the mixture a, when cooled, add the dispersion b which has been boiled separately. When cooled, add c.<sup>49</sup>

## FORMULA No. 50.

# QUICK BLACK

Soft Water	8	gal.	Caustic Potash	1/2	lb.
Castile Soap	11/2	ĺb.	Nigrosine 50	8	1b.
Grav Carnauba Wax	61/2	1b.			

<sup>47</sup> Ibid.

<sup>48</sup> Thid

<sup>49</sup> Bennett, Chem. Formulary, Vol. 4, p. 432 (1939).

<sup>50</sup> Davidsohn & Davidsohn, Shoe Creams & Polishing Waxes.

FORMULA No. 51.

## QUICK BLACK

Prepare 4 gals. of tragancanth solution by dissolving 2 lb. of the powdered gum in 4 gal. water.

Water Tragacanth Solution	-	gal. gal.	Nigrosine Metanil Yellow Dyestuff	15½ 6	
Caustic Soda	21/2	lb.	Ammonia		
Castile Soap	1	1b.	(added when cold)51	8	oz.
Gray Carnauba Wax					

FORMULA No. '52.

# QUICK BLACK

Soft Water	/- U	Nigrosine	12 lb.
Ammonium Linoleate	1⅓ lb.	Ammonia <sup>c</sup>	3 oz.
Yellow Carnauba Wax	1ь.		

FORMULA No. 53.

# QUICK BLACK

Prepare a casein solution by dissolving  $2\frac{1}{2}$  lb. casein in 2 gals. of hot water with the aid of  $3\frac{1}{2}$  oz. ammonia (.880).

(a) Water	3 gal.	(b) Water	4 gal.
Metanil Yellow	¼ lb.	Castile Soap	1¼ lb.
Nigrosine	8½ lb.	Gray Carnauba Wax	7 lb.
Casein Solution	2 gal.		

The nigrosine-casein mixture is prepared separately and allowed to become quite cold. The wax is boiled until in solution and the cold dye added quickly to cool it down. Stirring is continued until the whole is quite cold.<sup>53</sup>

<sup>51</sup> Ibid.

<sup>52</sup> Ibid.

<sup>53</sup> Davidsohn & Davidsohn, Shoe Creams & Polishing Waxes.

## FORMULA No. 54.

# QUICK BLACK

(a) Water	6	gal.	(b) Water	$4\frac{1}{2}$	gal.
Borax	11/4	1b.	Tragacanth Solution	1	gal.
Garnet Shellac	5	1b.	Castile Soap	13/4	1b.
Metanil Yellow	6	oz.	Gray Carnauba Wax	12	lb.
Nigrosine	14	1b.	Ammonia		
-			(added cold)	4	oz.

This ink is prepared with two solutions, the cold dye being added to the boiling wax to cool it down.<sup>54</sup>

#### FORMULA No. 55.

## ONE SETTING EDGE INK

Water	123/4	gal.	Gray Carnauba Wax		
Borax	1	Īb.	Tragacanth Solution	61/4	gal.
Bleached Shellac	41/2	1b.	Metanil Yellow	1/4	1b.
Caustic Soda	21/2	1b.	Nigrosine	18	1b.
Castile Soap	11/4	1b.	_		

Boil the ingredients together in the order indicated, and cool down rapidly with continuous agitation. When cold add

Aniseed Oil, dissolved in 11/4 oz. Methylated Spirits 55 1/2 gal.

## FORMULA No. 56.

#### HEAVY INK FOR SPONGY LEATHERS

(a) Water	4	gal.	(b) Water	4	gal.
Metanil Yellow	5	oz.	Tragacanth Solution	1	gal.
Nigrosine	111/4	1b.	Castile Soap	2	1b.
Casein Solution (21/2			Gray Carnauba Wax	101/2	1b.
lbs. to 2 gallons)	1.1/2	gal.			

The two solutions are prepared separately and the cold dye solution added to the emulsified wax.<sup>56</sup>

<sup>54</sup> Ibid.

<sup>55</sup> Ibid

<sup>&</sup>lt;sup>56</sup> Davidsohn & Davidsohn, Shoe Creams & Polishing Waxes.

## FORMULA No. 57.

# QUICK BLACK

(a) Soft Water	25 gal.	(b) Soft Water	10 gal.
Borax	2 lb.	Ethanolamine	2 lb.
Garnet Shellac	8 lb.	Stearic Acid	6 lb.
Nigrosine	40 lb.	Yellow Carnauba Wax	30 lb.
Nigrosine	40 lb.	Yellow Carnauba Wax	30 lb.
Metanil Yellow	2 lb.	Black Montan Wax 57	2 lb.

## FORMULA No. 58.

## BURNISHING INK

Paraffin Wax	3.0	Nigrosin, Water-Soluble	2.4
Carnauba Wax	3.0	Soda Ash, Calcined	3.0
Japan Wax	6.0	Water 58	76.6
Rosin	6.0		

## FORMULA No. 59.

## GLOSS INK

a. Montan Wax, Crude	15	b. Shellac	20
Colophony	2	Borax	7
Soft Paraffin 40/42°C.	3	Water	75
Potash Carbonate	1/2	c. Nigrosine NTL	51/2
Marseilles Soap	4	Water	25
Water	65		

Make up separately a, b, and c, and mix these three components together. <sup>59</sup>

## WHITE SHOE POLISHES

## FORMULA NO. 1.

Titanium Dioxide (Water		Japan Wax	1.0
dispersing grade)	15.0	Oleic Acid	0.5
Pine Oil	1.0	Triethanolamine	0.5
Sodium Alginate	0.3	Water	<b>7</b> 9. <b>7</b>
Gum Arabic	1.0	Preservative	Ŋ.S.
Carnauha Wax	1.0		•

The pine oil is emulsified with a small portion of the oleic acid and triethanolamine and the pigment wet thoroughly

<sup>57</sup> Ibid.

<sup>&</sup>lt;sup>58</sup> Bennett, Chem. Formulary, Vol. 4, p. 183 (1939).

<sup>&</sup>lt;sup>59</sup> Davidsohn & Davidsohn, Shoe Creams & Polishing Waxes.

with this emulsion. The sodium alginate is dissolved in a small part of the water and added to the pigment mix. The gum arabic is dissolved by heating to about 180°F. (on a water bath), with a small amount of water. When the gum arabic is completely dissolved, it is cooled to room temperature and added to the pigment mix. The waxes are heated with the remaining amounts of oleic acid and triethanolamine to the melting point (about 185°F.) of the carnauba wax and then a sufficient amount of water (boiling hot) is added while stirring vigorously. Stirring should be continued for about ten minutes or until a smooth emulsion is formed. This emulsion is cooled to room temperature with slight agitation.<sup>1</sup>

# Formula No. 2.

# WHITE BOTTOM GLOSS

Yellow Carnauba Wax	3½ 1	lb.	White Spirit	1/2	gal.
Beeswax	11/4 1	lb.	Ethanolamine	3/4	lb.
Japan Wax	1 1	lb.	Stearic Acid	11/2	1b.
Ceresine Wax	1/2 1	lb.	Water	1	gal.
Turpentine	1/2 8	gal.			

The waxes, stearic acid and the ethanolamine are melted together in a steam jacketed pan and the mixture of turpentine and white spirit added slowly so that there is no precipitation of the waxes. The water is boiled and stirred into the other melted components.<sup>2</sup>

# FORMULA No. 3.

#### WHITE GLOSS

Soft Water	½ gal.	Turpentine	½ gal.
Castile Soap	3/4 lb.	Raw Linseed Oil or	
Yellow Carnauba Wax	3 lb.	Mineral Oil	½ pt.

The water and soap are boiled together until a clear solution is obtained; the wax is melted in the second pan and

<sup>&</sup>lt;sup>1</sup> John, White Shoe Dressings and Cleaners (1940).

<sup>&</sup>lt;sup>2</sup> John, White Shoe Dressings & Cleaners (1940).

the turpentine and oil added to it. The wax solution is then rapidly transferred to the boiling water with constant stirring. The whole rapidly thickens to a creamy paste, which when cold is packed into one pound tins by the aid of a flat spatula. To obtain a smooth lustrous appearance the tin is then passed under a flat hot plate.<sup>3</sup>

#### FORMULA No. 4.

## WHITE KID CLEANER

a.	Carnauba Wax, Gray	50 g.	b. Turpentine	150 cc.
	Montan Wax, Bleached	50 g.	Benzene	100 cc.
	Paraffin Wax (50/52°C.)	20 g.	Trichlorethylene	150 cc.
	Japan Wax	20 g.	Acetone	50 cc.
	Rosin	15 g.		
	Diglycol Stearate	10 g.		
	Potassium Carbonate	32 g.		
	Water	600 g.		

Boil a together with stirring, until a homogeneous emulsion has been formed. Cool to  $40^{\circ}$ C., and add the solvents b. Stir until cooled.

#### FORMULA NO. 5.

## WHITE SHOE CLEANER

Titanium Dioxide	5	Petroleum Naphtha	13
Lithopone	15	Triethanolamine Stearate	2
Danmar	4	Water	51
Carbon Tetrachloride	10		

Danmar resin is dissolved in the petroleum naphtha and the solution is strained to remove undissolved matter. The triethanolamine stearate is dissolved in warm water. After cooling, the resin solution is stirred into the water solution, using vigorous agitation because an emulsion is being made and additions must be slow. When the emulsion is smooth,

<sup>3</sup> Ibid.

<sup>&</sup>lt;sup>4</sup> Bennett, Chem. Formulary, Vol. 4, p. 428 (1939).

the carbon tetrachloride is stirred in followed by the pigments.<sup>5</sup>

#### METAL POLISHES

#### FORMULA No. 1.

2 parts of fatty acids from tallow or from hardened fat, 8 parts of talloil, 5 of petroleum and 5 of denatured alcohol are treated with 5 parts of ammonia, sp. gr. 0.91, for saponification of the fatty acids. To this is added 45 parts of water and then a mixture of 5 parts of bentonite and 25 parts of amorphous silica. This product is rather viscous and settles out only slowly.<sup>1</sup>

#### FORMULA No. 2.

Gum Tragacanth	0.20	Glycerin	5.65
Water	63.15	Diatomaceous Earth	12.25
Alkali Metal Salt of a		Neutral Oil (Mineral)	17.00
Sulphated Alcohol Having		Amyl Acetate	0.25
More Than 8 Carbon		Formaldehyde 2	1.00
Atoms (Wetanol)	0.50	-	

#### FORMULA No. 3.

Kieselguhr	5.0	Red Oil	2.0
Silica Dust	4.0	Triethanolamine	0.5
Ferric Oxide	4.0	Ammonia (26° Bé.)	1.5
Naphtha Kerosene	19.0 10.0	Water	54.0

The oleic acid is first mixed with the kerosene and naphtha, the abrasives then being stirred in. Stirring is continued while the ammonia and triethanolamine solution is added. If desired, the proportion of water may be increased by about another 10%.

<sup>&</sup>lt;sup>5</sup> Bennett, Chem. Formulary, Vol. 5, p. 535 (1941).

<sup>&</sup>lt;sup>1</sup> Ibid., p. 526.

<sup>2</sup> P.C.

<sup>&</sup>lt;sup>3</sup> Bennett, Chem. Formulary, Vol. 5, p. 526

#### FORMULA No. 4.

Naphtha Oleic Acid Abrasive	62 1 7	Triethanolamine Ammonia (26°) Water	1 128

#### FORMULA No. 5.

Add the mixture b to the solution a with agitation. Work in c.

Let stand overnight. Strain.5

## FORMULA No. 6.

1.	Naphtha	60	4. Ammonia	2-3
	Oleic Acid Distilled	5	5. Clay	1
2.	Triethanolamine	2	Tripoli	11
	Water	125	Aluminum Oxide	77
3.	Naphtha	5		

To the solution 1 add with good agitation the solution 2. When emulsified, add 3, 4, and finally the mixture 5 to get a paste.<sup>6</sup>

## FORMULA No. 7.

a. Soft Soap	30	c. Kieselguhr	20
b. Turpentine	10	Emery	20
c. Alcohol	5	Chrome Oxide	5
d. Water	10		

Thin a with b, c, d in the given order; work in  $e^{-\tau}$ 

## FORMULA No. 8.

a. Oleic Acid or Talloil	5	d. Whiting	10
Turpentine	15	Emery	20
b. Ammonia	3	Glass, Powdered	10
c. Water	30	Graphite Dust	7

<sup>4</sup> Ibid., Vol. 1, p. 426 (1933).

<sup>&</sup>lt;sup>t</sup> Ibid., Vol. 4, p. 412 (1939).

<sup>€</sup> Ibid., p. 437.

Bennett, Chem. Formulary, Vol. 4, p. 409 (1939).

To the solution a add b with good agitation. Thin with c. Work in d to get a paste.<sup>8</sup>

## FORMULA No. 9.

Water	480.0	Silica	180.0
Suspendite	15.0	Solvent Naphtha	13.6
Ammonium Laurate S	40.0	Ammonia (26°)	22,4

The Suspendite is added to warm water with stirring until smooth. The ammonium laurate S is added with stirring and then the silica. Under rapid agitation this should take about 10–15 minutes. Then add the naphtha with continued stirring and finally the ammonia.

The consistency of the polish can be varied by varying the amounts of the different ingredients.9

## FORMULA No. 10.

Ammonium Linoleate R.	20	Naphtha V. M. & P.	10
Oxalic Acid	2	Silica	60
Ammonia	4	Alcohol	5
Water	150		

Heat the water to 70°C. Dissolve the oxalic acid, add the silica and stir until dispersed. Add the ammonia and stir for a few minutes keeping at 70°C. Then add the ammonium linoleate R. and stir until dissolved. Add the naphtha slowly and stir. The mixture becomes very heavy. The alcohol is then added slowly until the desired body and viscosity are obtained.<sup>10</sup>

#### FORMULA NO. 11.

Ammonium Linoleate R.	20	Alcohol
Naphtha V. M. & P.	100	Ammonia
Silica	40	

Heat the naphtha cautiously to 60°C. Dissolve the ammonium linoleate R. and disperse the silica. Add the ammonia

<sup>8</sup> Ibid.

<sup>9</sup> Ibid., Vol. 5, p. 526 (1941).

<sup>10</sup> Ibid., Vol. 4, p. 408 (1939).

when the mixture becomes very heavy. It is then thinned down as desired by the addition of the alcohol.<sup>11</sup>

## FORMULA No. 12.

a. Ammonium Linoleate	9	c. Whiting	40
Turpentine	20	Chrome Oxide	5
b. Alcohol	4		
Water	22		

Mix a, which has been previously heated to about 50°C. with b, adding the latter with good agitation; work in c.<sup>12</sup>

## FORMULA No. 13.

a. Soft Soap	20	b. Water	15
Turpentine	15	c. Whiting	35
Alcohol	5	Yellow Ochre	10

Make solution a, add b, and when homogeneous, use to make a paste with c.<sup>13</sup>

#### FORMULA No. 14.

Tripoli	1¼ lb.	Sodium Hydroxide		
Pine Oil (Steam		(Sticks)	1/2	oz.
Distilled)	$1\frac{1}{2}$ pt.	Water	3	pt.
Oleic Acid	7 fl. o2.			_

Mix the pine oil and oleic acid. Dissolve the sodium hydroxide in the water and run this mixture into the oil while mixing. Mixing should be continued for about 5 minutes. The tripoli is finally added and thoroughly mixed in. This metal polish is applied with a soft cloth.<sup>14</sup>

## FORMULA No. 15.

Stearic Acid	10	Alcohol	
Naphtha	30	Mixture of Clay.	
Caustic Potash (30° Bé.)	5		To make paste

<sup>11</sup> Bennett, Chem. Formulary, Vol. 4, p. 409 (1939).

<sup>12</sup> Ibid.

<sup>18</sup> Thid.

<sup>14</sup> Crcwley, Money Making Formulas.

To the naphtha-stearic acid solution add the alkali, then the alcohol. Work into a paste with the minerals.15

## FORMULA No. 16.

Potassium Hydroxide	40 g.	Alcohol	25 cc.
Water	900 cc.	Ethylene Dichloride	50 cc.
Olive Elaine	150 cc.	-	

Add the potassium hydroxide to the water, warm to 75°C. and slowly stir in the olive Elaine until completely dissolved. Cool and add the alcohol and ethylene dichloride.16

## FORMULA No. 17.

1. Ortho Dichlorbenzol	5	Tripoli or Silex	50-75
Naphtha or Mineral Spirits	20	Suspendite	9
Pine Oil	4	Water	260
2. Trihydroxyethylamine		3. Ammonium Hydroxide	12
Linoleate	2	·	

Add "1" to "2" with stirring and then stir in "3"; allow to stand overnight and stir before packaging.

This gives a polish which does not separate if made properly. If a thicker polish or paste is desired the Tripoli is increased and the liquids decreased.17

#### FORMULA No. 18.

Dissolve 5 parts curd soap in 40 parts water, adding 20 parts of a 5 per cent methyl cellulose slime. Then add 15 parts kieselguhr, 10 parts tripoli, and 10 parts denatured alcohol.<sup>18</sup>

#### FORMULA No. 19.

Tripoli	20.0	Yarmor	25.0
Oleic Acid	7.0	Water 19	47.5
Sodium Hydroxide (100%)	.5		

<sup>&</sup>lt;sup>15</sup> Bennett, Chem. Formulary, Vol. 4, p. 411 (1939).

<sup>&</sup>lt;sup>16</sup> Ibid., Vol. 3, p. 284 (1936).
<sup>17</sup> Ibid., Vol. 1, p. 426 (1933).

<sup>18</sup> P. C.

<sup>&</sup>lt;sup>19</sup> Ibid., Vol. 1, p. 425 (1933).

#### FORMULA No. 20.

a. Ammonium I	Linoleate	6	c. Calcium Carbonate	40
Benzine		16	Pigment (e.g. Ultramarine)	10
b. Water		28		

Make emulsion by adding b to a with stirring. Work into this the fine powder c.

Nos. 1 and 2 are for tin cans, No. 3 is for collapsible tubes.20

## FORMULA No. 21.

a. Ammonium Linoleate	9	b. Emery	20
Turpentine Substitute	16	Whiting	25
Alcohol	2	Glass Powder	10
		c. Water	18

Mix a, and wet thoroughly b in this oil-solvent mixture. Add c to the whole with thorough agitation and in small portions.<sup>21</sup>

## FORMULA No. 22.

a. Ammonium Linoleate	25	b. Whiting	15
Water	40	Quartz Flour	10
		Chromium Oxide 22	10

#### FORMULA No. 23.

a. Soft Soap	15	d. Green Earth	10
Methyl Cyclohexanol	2	· Whiting	10
b. Turpentine	15	Pumice Meal	10
c. Ammonia (0.910)	5	Emery	10
Water	23	-	

Stir together a and b, add c with good agitation. Now add the powders d, mixing very thoroughly.<sup>23</sup>

## FORMULA No. 24.

a. Oleic Acid	15	b. Ammonia (0.910)	5
Mineral Oil, White Alcohol	10 45	c. Chrome Oxide	25

<sup>&</sup>lt;sup>20</sup> Bennett, Chem. Formulary, Vol. 4, p. 413 (1939).

<sup>&</sup>lt;sup>21</sup> Ibid., p. 412.

<sup>22</sup> Ibid.

<sup>23</sup> Ibid.

Emulsify adding b to a with stirring. Work in c with thorough agitation.<sup>24</sup>

## FORMULA No. 25.

a. Diglycol Stearate	6	c. Kieselguhr	14
Water	66	Iron Oxide Red	6
b. Oleic Acid	5	d. Ammonia (0.910)	3

Mix b with the hot solution a. Work in the abrasives c, and saponify with d. Mix thoroughly, and allow to stand overnight. Strain.<sup>25</sup>

#### FORMULA No. 26.

a. Diglycol Laurate	8	c. Kieselguhr, Calcined	20
Water	50	Lime Green	4
b. Oleic Acid or Talloil,		d. Ammonia (0.910)	4
Distilled	4	e. Alcohol	10

Mix b with a, work in c with good agitation; saponify with d. Let stand overnight. Keep covered.

Add e. Strain 6 hours later.26

#### FORMULA No. 27.

# PROTECTIVE COATING FOR ALUMINUM AND OTHER METALS

Paraffin Wax	4	Flexo Wax C	2
Diglycol Stearate	1	(Amorphous Petroleum Wa	ax)
		Water	40

Heat together and mix with high speed agitator until uniform emulsion is formed.

Apply by spraying or brushing.27

<sup>24</sup> Thid.

<sup>&</sup>lt;sup>25</sup> Bennett, Chem. Formulary, Vol. 4, p. 412 (1939).

<sup>26</sup> Thid

<sup>&</sup>lt;sup>27</sup> Ibid., Vol. 5, p. 335 (1941).

## Practical Emulsions

## STOVE POLISHES

## FORMULA No. 1.

Cumarone Resin Paraffin Wax, Soft Montan Wax, Crude Potash Carbonate		Water Nigrosin Base BB Lamp Black Graphite <sup>1</sup>	200 10 24 56
F	ORMULA	No. 2.	
Crude Montan Wax Rosin	2 1	Carnauba Wax	2
Heat to 90°C. with sti	rring	and to it add slowly	
Caustic Potash Water (Boiling)	2 86	Nigrosine	3
Keep on heat and agi	tate v	igorously until uniform.	Cool
Graphite Flake	5	Lampblack	3
Mix thoroughly until	unifor	rm.²	
Ŧ	ORMUL	A No. 3.	
Paraffin Wax (40°-42°C.) Ozokerite, Crude Turpentine Rosin (D to K Grades) Lamp Black		Ammonium Linoleate Water Nigrosine, Oil-Soluble	24.0 4.0 20.0 0.8 1.0

The oil and waxes are carefully melted. The potash and nigrosine are dissolved in the water and gradually stirred into the wax mixture at 85° to 90°C. The resultant product

FORMULA No. 4.

2

2

82

Caustic Potash

Nigrosine

Graphite

Lampblack

Rosin Oil

Water

Montan Wax (Black)

Carnauba Wax

<sup>&</sup>lt;sup>1</sup> Bennett, Chem. Formulary, Vol. 5, p. 531 (1941).

<sup>&</sup>lt;sup>2</sup> Ibid., Vol. 4, p. 418 (1939).

<sup>\*</sup> Ibid., Vol. 5, p. 531 (1941).

is agitated thoroughly during the cooling, when the graphite and lampblack are uniformly incorporated. The caustic potash forms a soap with the resin and aids emulsification and retards settling of the black.<sup>4</sup>

## FORMULA No. 5.

Stearic Acid Spindle Oil	10 g. 20 g.	Tetralin Abrasive (Emery, Tripoli,	10 g.
Triethanolamine Linoleate	27 g.	Silicaceous Earth,	
Water	13 g.	etc.) unti	il pasty
Spindle Oil	20 g.		

Dissolve the fatty acids in the first spindle oil, mix with triethanolamine linoleate; add the second spindle oil, the tetralin, and ultimately the abrasive.<sup>5</sup>

## FORMULA No. 6.

a. Graphite	24	b. Hard Soap	3.2
Glycerin, Crude	5	Water	26
Formaldehyde	1	Paraffin	4

Prepare the solution b by boiling, and add to it the paste a. Stir till cold, and pour.<sup>6</sup>

#### FORMULA No. 7.

a. Carnauba Wax	6	Diglycol Stearate	1
Montan Wax, Bleached	3	Potash Carbonate	1
Beeswax	3	Water	45
Rosin	1	b. Turpentine	40

Boil a until a homogeneous mass is formed. Thin with b.

#### FORMULA No. 8.

a. Montan Wax, Crude	2.5	c. Potassium Carbonate	1.5
Rosin	0.8	Water	53.9
Japan Wax	0.7	d. Graphite	28.0
b. Nigrosin, Oil Soluble	0.6	Soot	12.0

<sup>4</sup> P. C.

<sup>&</sup>lt;sup>5</sup> Bennett, Chem. Formulary, Vol. 4, p. 416 (1939).

<sup>&</sup>lt;sup>6</sup> P. C.

<sup>7</sup> Ibid., Vol. 4, p. 416 (1939).

Melt a. Dissolve b in the melted a.

Boil c, and stir in d to get a smooth suspension.

Mix c + d into a + b with good stirring. Put into cans while hot.<sup>8</sup>

## FORMULA No. 9.

a.	Crude Montan Wax	2 lb.	b. Nigrosin,	Water-Soluble	1 lb.
	Carnauba Wax	2 lb.	Gra		20 lb.
	Rosin	1 lb.			
	Potash Carbonate	2 lb.			
	Water	72 lb.			

Boil a until homogeneous. Stir in b gradually, until smooth.9

## MISCELLANEOUS POLISHING EMULSIONS

#### FORMULA No. 1.

## TYPE "SIDOL"

Oleic Acid, Distilled	20	Neuburg Chalk	18
Alcohol	15	Kieselguhr	12
Heavy Benzine	15	White Bolus	
Ammonia (0.91)	12		

The oleic acid, dissolved in the solvent mixture, is saponified on the water-bath at temperature below the boiling point of the alcohol (60–70°C.). The strongly smelling liquid is now thickened with the abrasives which are added with good agitation.<sup>1</sup>

#### FORMULA No. 2.

a. Neuburg Chalk	80.0	b. Oleic Acid	3.5
Kieselguhr	4.0	Ammonia (0.91)	1.0
Water	300.0	Alcohol	6.5
		Naphtha	10.0

<sup>8</sup> P. C.

<sup>&</sup>lt;sup>9</sup> Ibid., Vol. 4, p. 416 (1939).

<sup>&</sup>lt;sup>1</sup> Bennett, Chem. Formulary, Vol. 4, p. 408 (1939).

To the mixture a, made 24 hours earlier and left overnight, add b, which is made by saponification at gentle heat to a soft-soap-like mass.<sup>2</sup>

## FORMULA No. 3.

Water	110	Turpentine	7½
Oxalic Acid	16	Neuburg Chalk	125
Oleic Acid	25	Water	215
Stearic Acid	4	Ammonia (0.91)	27½
Alcohol	15	Ammonia (0.91)	211/2

Dissolve the oxalic acid in  $\frac{1}{3}$  of the necessary water by heating to about 60°C. and add the remainder of the water.

Melt up the fatty acids, turn out any flames, and add the solvents with stirring.

Add the oils to the water, with stirring, add the chalk, and ultimately saponify with the ammonia, until, after an initial thickening, the mass starts to go thin again.

Keep in kettle for 2 days, stirring intermittently. If the mass still settles, add more oleic acid.<sup>3</sup>

#### FORMULA NO. 4.

a. Olein. Distilled	9	c. Oxalic Acid	2
Steaic Acid	2	Water (50-60°C.)	70
Alcohol	10	d. Neuburg Chalk, Fine	25
b. Ammonia (0.91)	14		

Dissolve a by heating to about 50–60°C. on water bath, saponify with b, and add the solution c. The resulting liquid is mixed with d in a good mixer.

#### FORMULA NO. 5.

Oleic Acid	16	White Spirit	10
Ammonia	10	Oxalic Acid	3
Clay, Powdered	60	Water 5	160
Alcohol	10		

<sup>&</sup>lt;sup>2</sup> Ibid.

<sup>3</sup> Ibid.

<sup>4</sup> Bennett, Chem. Formulary, Vol. 4, p. 408 (1939).

<sup>5</sup> Ibid.

#### FORMULA No. 6. Ammonia 6 Oleic Acid 18 White Spirit 150 Alcohol 6 Clay, Powdered 56 FORMULA No. 7. Spindle Oil 4 Ozokerite 4 Oleic Acid 10 Clav 15 White Spirit 1/2 3 Ammonia 7

## FORMULA No. 8.

a. Ammonium Oleate	2.1	c. Ammonia (0.91)	0.5
Water	19.5	d. Alcohol	2.1
b. Stearic Acid	1.0	e. Neuburg Chalk 8	6.0

Heat a to almost a boil to dissolve; add b with good stirring, cool to 75°C., and saponify with c. Cool to 50°C., add d and work in e with very thorough mixing.

	FORMULA	No. 9.	
Water Bentonite Naphthenic Soap	60 1 6	Silica or Tripoli Mineral Oil 9	15 18
	FORMULA	No. 10.	
Gray Carnauba Wax Japan Wax	25 5	Rosin	5
Melt and stir in			
Turpentine Substitute	60		
Strain and add			
Solution of Potash (1%)1	5		

<sup>6</sup> Ibid.

<sup>7</sup> Ibid.

<sup>8</sup> Ibid.

<sup>9</sup> Ibid.

<sup>10</sup> P. C.

## FORMULA No. 11.

a. Ammonium Linoleate		c. Graphite	20
Water	60	Lampblack	5
b. Water	5	-	
Potato Flour	2		

Make solution a, and separately paste b. Add b to a, mixing thoroughly. Work c slowly into this dispersion.<sup>11</sup>

## FORMULA No. 12.

Carnauba Wax	4989	Diglycol Laurate	200
Solvent Naphtha	7000	Triethanolamine	450
Stearic Acid	1360	Water	29937
Nigrosin, Oil Soluble	907	Nigrosin, Water Soluble	453

Dissolve the water-soluble nigrosin in the water, add the triethanolamine, and, while hot, the stearic acid and diglycol laurate. Take up to a boil.

Add to this the solution of carnauba wax and the oil-soluble nigrosin in the naphtha, at 85-90°C. Stir until cold.

#### POLISH POWDER

Kerosene	20 fl. oz.	Mineral Oil, Light	10 fl. oz.
Silica (300 Mesh)	2 oz.	Sulfonated Castor Oil	4 fl. oz.
Infusorial Earth	1 lb.		

Mix the oils and powders separately and work the two components together. Four ounces of this mass are to be stirred into a quart of water for use. On standing, the water solution will settle out. Shaking will restore it to a usable state <sup>1</sup>

#### FUR GLAZE

Add 10 parts of ammonium linoleate to 500 parts of water and to this mixture is added 50 parts of melted carnauba wax.<sup>2</sup>

<sup>11</sup> P. C.

<sup>&</sup>lt;sup>12</sup> Bennett, Chem. Formulary, Vol. 4, p. 430 (1939).

<sup>&</sup>lt;sup>1</sup> Crowley, Money Making Formulas.

<sup>&</sup>lt;sup>2</sup> Bennett, Chem. Formulary, Vol. 2, p. 297 (1935).

## NEATSFOOT OIL POLISH

Neatsfoot Oil (20° Cold Test)	50	Soap Chips	8
Lanolin Anhydrous	35	Water 3	90
Japan Wax	20		

#### GLASS POLISH

1. Ammonium Linoleate	20	3. Water	200
2. Orthodichlorbenzol	100	4. Infusorial Earth	60

Dissolve (1) in (3) overnight and run (2) in while beating with high speed mixer. Then beat (4) in until uniform.<sup>3a</sup>

## OIL POLISH

Mineral Oil	60	Triethanolamine	4
Naphtha	26	Methanol	4
Turpentine	3	Water 4	120
Stearic Acid	9		

## ABRASIVE POLISH

1. Carnauba Wax	42 lb.	6. Triethanolamine	8 oz.
1. Califauba Wax	<del>4</del> 2 10.	O. IIICIIIAIIOIAIIIIIC	0 02.
2. Beeswax	18 lb.	7. Stearic Acid	2 lb.
3. Ceresin	18 lb.	8. Tripoli	24 lb.
4. Varnolene	3 gal.	9. Pumice	15 lb.
5. Water	3 gal.		

Melt 1, 2, 3, 7 with 4. Heat 5 and 6 to 90°C., add to wax mixture and stir till emulsified. Then add 8 and 9 and stir till cool.<sup>5</sup>

<sup>&</sup>lt;sup>3</sup> Ibid., Vol. 1, p. 425 (1933).

<sup>&</sup>lt;sup>3a</sup> Ibid., p. 424.

<sup>4</sup> Ibid., p. 427.

<sup>5</sup> P. C.

## CHAPTER XVII

# RESIN AND RUBBER EMULSIONS

## ROSIN EMULSIONS

150

FORMULA No. 1.

Rosin 700 Glue Water 2100

Melt glue in water and while boiling hot, slowly add melted rosin, agitating violently. Continue agitation until perfectly smooth.<sup>1</sup>

FORMULA No. 2.

 Rosin
 700
 Gelatin
 150

 Water
 2100

Melt gelatine in water and while boiling hot, add melted rosin slowly, agitating violently. Continue agitation until perfectly smooth.<sup>2</sup>

## FORMULA No. 3.

Rosin	700	Stearic Acid	63
Water	2100	Triethanolamine	21

Melt rosin and stearic acid together. Acid triethanolamine to water. Heat water to boiling point and stir in melted rosin. Stir until smooth <sup>3</sup>

<sup>&</sup>lt;sup>1</sup> Bennett, Chem. Formulary, Vol. 1, p. 155 (1933).

<sup>&</sup>lt;sup>2</sup> Ibid.

<sup>&</sup>lt;sup>3</sup> Ibid.

FORMULA No. 4.

Rosin 14 Water

Heat to boiling; turn off heat and add while stirring vigorously

Ammonium Hydroxide

5 Water

34

84

Stir until all lumps disappear (reheating may be necessary).4

## FORMULA No. 5.

1. Rosin	100 100	4. Ammonium Hydroxide 5. Water	200
2. Naphtha	100	J. Walti	200
3 Ammonium Linoleate	3		

Heat 1 to 150°C. and turn off flame; run 2 (which has been previously heated on a water-bath to 90°-100°C.) into it slowly and stirring until all rosin has dissolved; cool and add 3, 4, and 5 mixed together slowly with vigorous stirring.<sup>5</sup>

#### FORMULA No. 6.

Caustic Soda		1b.	Rosin	1b.
Water	100	lb.		

Boil together and stir until uniform.6

#### FORMULA No. 7.

# ROSIN, TURPENTINE EMULSION

Rosin	11.0	Water	50.0
Turpentine	2.5	Ammonia	15.0
Ammonium Linoleate	2.0		

The ammonium linoleate and water are taken up in the usual way; heated, and mechanically agitated (high-speed mixer). The rosin and turpentine are heated together and added to the ammonium linoleate dispersion in water to

6 P. C.

<sup>4</sup> Ibid.

<sup>&</sup>lt;sup>5</sup> Ibid., Vol. 1, p. 443 (1933).

which has previously been added the 15 cc. of ammonia. Stirring is continued until cool.

This gives a paste emulsion.7

## FORMULA No. 8.

## RESIN EMULSION

Rosin, Ester Gum, Cumar, or		Triethanolamine	2 oz.
Other Resin, (Melted)	3 lb.	Casein Solution	2 lb.
Oleic Acid	1 oz.	Water, To make 8	1 gal.

#### FORMULA No. 9.

#### PARAFFIN ROSIN EMULSIONS

In producing these dispersions, various proportions of paraffin and rosin may be used, for instance, from as high as 85% paraffin and as low as 15% rosin to as low as 15% paraffin and as high as 85% rosin. Assuming that a mixture of about 50% paraffin and about 50% rosin is employed, the mixture is melted and heated to about 220°F., which temperature is materially above the melting point of the paraffin (130°F.) and above the melting point of the rosin (about 180°F.). The heat-liquefied thermoplastic mixture is then commingled as a regulated stream flowing, say, at the rate of 15 pounds per minute with a regulated stream of 5% caustic soda solution at 110°F., flowing, say, at the rate of 20 pounds per minute. Under these conditions, an aqueous dispersion of a solids content of about 45% and at a temperature of about 155°F. is produced, which when suddenly chilled to a temperature below 130°F. to prevent coalescence of dispersed particles, is of a creamy consistency. In producing asphalt dispersions, at least about 35% rosin, based on the total weight of thermoplastic material, should be used in order to produce a stable dispersion of fine particle size.

Ibid., Vol. 1, p. 163 (1933).

<sup>&</sup>lt;sup>3</sup> Ibid., Vol. 2, p. 189 (1935).

Assuming that a mixture of about 50% asphalt having a melting point of about 150°F. and about 50% rosin is employed, the mixture should be heated to about 300°F., whereupon it is brought in contact with a caustic soda solution under practically the same conditions as in the case of the paraffin dispersion, except that the solution should be at 150° to 160°F. The resulting dispersion has a temperature of about 200°F., and when permitted to cool, is of a smooth, soft, paste-like consistency.

#### FORMULA No. 10.

## "CUMAR" EMULSION

Naphtha 20 Cumar 20

Heat above until dissolved; while at 90–100°C. add following solution (at 90–100°C.) slowly with vigorous stirring.

Ammonium Linoleate Paste 10 Water 10 40

#### FORMULA No. 11.

#### GELVA RESIN EMULSION

Polyvinylacetate Resin		Water	39.4
(Gelva)	25.0	Sodium Lauryl Sulfate	0.2
Toluol	55.0	Sulfonated Castor Oil 11	0.4
Butyl Acetate	20.0		

#### ETHYL CELLULOSE EMULSIONS

	FORMULA	No. 12	No. 13	No. 14
	Ethyl Cellulose	5.0	5.0	5.0
	Octyl Acetate	63.0	5.0	5.0
1	Toluol	63.0	63.0	63.0
	Diphenyl Oxide	0.6	1.4	1.0
	Dow Plasticizer No. 6	1.4	0.6	1.0
2	Deceresol O T	2.0	2.0	2.0
۷. ۱	Water	13.012	13.013	

<sup>&</sup>lt;sup>9</sup> Bennett, Chem. Formulary, Vol. 2, p. 189 (1935).

<sup>&</sup>lt;sup>10</sup> Ibid., p. 188.

<sup>&</sup>lt;sup>11</sup> Billing, U. S. 2,230,792 (1941).

<sup>&</sup>lt;sup>12</sup> Bennett, Chem. Formulary, Vol. 5, p. 73 (1941).

<sup>&</sup>lt;sup>13</sup> Ibid.

<sup>14</sup> Ibid.

Add 2 to 1 slowly while mixing vigorously with an electrical mixer.

#### METHYL METHACRYLATE EMULSIONS

#### FORMULA No. 15.

1. Monomeric Methacrylate	25	4. Water	64
2. Dibutyl Phthalate	5	5. Hydrogen Peroxide (30%)	3
3. Deceresol O. T. (10%)	4		

Mix 1 and 2 and add slowly to mixture of 3, 4, and 5, stirring well. Then pass through a colloid mill or homogenizer.<sup>15</sup>

#### FORMULA No. 16.

, Monomeric Methacrylate	12.5	3. Deceresol O. T. (10%)	4.0
1. Monomeric Styrene	12.5	4. Water	64.0
<ol> <li>Monomeric Methacrylate</li> <li>Monomeric Styrene</li> <li>Dibutyl Phthalate</li> </ol>	5.0	<ol> <li>Hydrogen Peroxide 16</li> </ol>	2.0

## FORMULA No. 17.

Heat to 80°C. and mix vigorously.16

# NATURAL RESIN EMULSIONS

#### FORMULA NO. 1.

Elemi	58	Oleic Acid	7
Batavia Dammar	14	Commercial Latex Solution	
Water	128	(About 36% Latex)	70
Triethanolamine	3		

Melt the elemi and stir in the dammar. Raise the temperature to 125°C. When the mixture is homogeneous, allow to cool and set the container into a boiling water bath. Add the oleic acid and then the triethanolamine with stirring. Add the water, pre-heated to boiling, portion by portion, stirring and incorporating after each small portion has been added

<sup>15</sup> Ibid.

<sup>16</sup> Ibid.

<sup>16</sup>a Ibid., p. 550.

before adding the next portion. The mixture thickens at first but as additions of water continue it changes to a thin white liquid. After all the water has been added, allow the mixture to cool but continue the stirring until it is cool. Add the latex solution with stirring.<sup>17</sup>

## FORMULA No. 2.

Manila DBB (Ground)	144	Commercial Latex Solution	
Water	206	(About 36% Latex)	3690
Ammonium Hydroxide	60		

Mix the Manila, water and ammonia until all the resin dissolves. Strain. To this solution, add the latex solution.<sup>18</sup>

#### FORMULA No. 3.

Manila DBB	144	Commercial Latex Solutio	n
Water	206	(About 36% Latex)19	1230 lb.
Ammonium Hydroxide	60		

Mix the Manila, water and ammonia until all the resin dissolves. Strain. To this solution, add the latex solution.

#### CELLULOSE ESTER EMULSIONS

A pyroxylin base is prepared by colloiding 12.5 parts by weight of alcohol-wetted pyroxylin (10 parts of dry ½" pyroxylin) with 20 parts by weight of blown linseed oil in a suitable mixer, such as the Werner and Pfleiderer mixer. 25 parts by weight of a solvent mixture are then added to the colloided mass in portions equalling 5 parts by weight to form a homogeneous base having the following composition:

Pyroxylin (½ sec.)	10	Butyl Acetate	20
Alcohol (Denatured)	2½	Butyl Lactate	5
Blown Linseed Oil	20	-	

<sup>17</sup> Amer. Gum Importers' Assn.

<sup>18</sup> Ibid.

<sup>19</sup> Thid

An emulsion is prepared by heating 0.5 part by weight of sodium oleate with 15 parts by weight of gasoline to a clear gel; after which 2 parts by weight of water are added to the hot gel with vigorous stirring, thus forming a concentrated emulsion of gasoline in water that is stabilized by sodium oleate. For convenience this will hereafter be called the agent emulsion.

The presolution or solvating of the sodium oleate in gasoline or some similar liquid is desirable to assure uniform distribution.

17.5 parts by weight of the agent emulsion are then stirred vigorously into 57.5 parts by weight of the pyroxylin base with a high speed stirrer of the propeller blade type.20

## PHENOL-FORMALDEHYDE RESIN EMULSION

Phenol-Formaldehyde Resin	45	Sulphonated Sperm Oil	5
Paraffin 'Oil	5	Olein	5
(Heat together)		Cyclohexanol	1

Partially saponify above with aqueous caustic soda then add

Glue Water 21/2 45

Mix in homogenizer or colloid mill.21

#### POLYSTYRENE RESIN EMULSION

ſ	Polystyrene Resin	7.5	[ Emulsifying	
	Toluol	22.5	Emulsifying Agent	1.0
ſ	Octyl Acetate	6.5	b. { (Deceresol O T)	
,			Diglycol Laurate	0.5
			b. { (Deceresol O T)     Diglycol Laurate     Water	12.5
			c. Toluol	50.0 g.

Dissolve a: to this add b slowly while mixing vigorously, then add c and stir until uniform.

This gives a stable film-forming emulsion.<sup>22</sup>

<sup>&</sup>lt;sup>20</sup> U. S. 1,970,572.

<sup>&</sup>lt;sup>21</sup> Ibid., Vol. 3, p. 109 (1936). <sup>22</sup> Ibid., Vol. 5, p. 72 (1941).

## "VINYLITE" EMULSIONS

One of the most satisfactory emulsifying agents is ammonium oleate, prepared "in situ" by adding oleic acid to the lacquer phase and an excess of ammonia to the water phase. Triisopropanolamine oleate prepared in a similar manner can be used if the volatility of ammonia is undesirable. Sodium lauryl sulfonate may also be used and this is dissolved entirely in the water phase. The amount of emulsifying agent required is of the order of 0.5 to 5 per cent of the weight of resin present, the exact amount varying for specific agents and specific types of emulsions.

A small amount (0.5 per cent of resin weight) of bentonite clay dispersed first in a little water and then added to the finished emulsion improves the stability upon storage appreciably. This should not be added to the water phase before emulsification, however. A ball mill is suggested as a suitable means of preparing the preliminary bentonite dispersion.

## PLASTICIZED VINYLITE RESIN AYAA EMULSION

Vinyl Resin AYAA	50.0	Oleic Acid	1.5
Tricresyl Phosphate	5.0	Distilled Water	92.0
Toluene	43.5	Aqua Ammonia 23	8.0

#### RUBBER EMULSIONS

# FORMULA No. 1.

Rubber Bentonite	70 1½	Water <sup>1</sup>	10¾
	Formula	No. 2.	

Sodium Solution	7	Bentonite	20
Sodium Silicate	58	Water 2	13

<sup>&</sup>lt;sup>23</sup> Carbide & Carbon Chemicals Corp., New York, N. Y. (Catalog). <sup>1, 2</sup> U. S. 1,498,387.

#### FORMULA No. 3.

A mixture of rubber 100, benzene 10–100, glue 1 and casein 1 part is masticated while slowly adding an aqueous solution of potassium oleate until the rubber constitutes the dispersed phase of the batch. The product is suitable for use as a cementing medium.<sup>3</sup>

## ARTIFICIAL LATEX

Reclaimed Rubber	48.5	Rosin Soap	9.0
Cumar, Mineral Rubber or		Whiting	40.0
Gum Rosin	2.5	Water as desired.	

The reclaimed rubber and softener are mixed together in a Banbury mixer for approximately 20 minutes. The whiting and rosin soap are made into a stiff putty-like paste with water. This paste is then placed in a Werner-Pfleiderer mixer and the rubber-softener blend added gradually to the paste in the mixer. This process will require 30 to 80 minutes, during which time the mixer should be heated with steam passing through the jacket to a temperature of approximately 180°F. The mixing is continued until the rubber is completely dispersed in the form of fine particles 2-20 mu. in diameter. The dispersion thus made can be diluted as desired with water to reduce the viscosity. Bodying agents such as gum tragacanth and curing ingredients may be added with the water. This dispersion may be used to produce a rug backing material by spreading it on the back of the rug and evaporating the water.4

#### RUBBER RESIN EMULSION

1. Rubber Resin	40	3. Water	150
2. Ammonium Linoleate			
Paste	10		

<sup>&</sup>lt;sup>3</sup> Bennett, Chem. Formulary, Vol. 1, p. 163 (1933).

<sup>4</sup> Ibid., Vol. 5, p. 543 (1941).

Heat 1 and 2 until homogeneous and add water to it slowly with vigorous mixing.5

#### FORMULA NO. 1.

#### CHLORINATED RUBBER EMULSIONS

Stable emulsions can easily be obtained without the use of emulsifying agents, if, for example, 70 g. of chlorinated rubber, 30 g. xylol and 40 g. of water—kept apart from each, i.e., the chlorinated rubber is not dissolved but fed simultaneously and progressively into a stirring device in operation.6

## FORMULA No. 2.

Casein	2.0	Turkey Red Oil	6.0
Ammonia (28%)	0.1	•	

Heat to 35°C. with stirring and add slowly a solution of Chlorinated Rubber 20

Trichlorethylene

80

Stir until uniform. This disperses well in water.

# CHLORINATED RUBBER EMULSION

20 parts, by weight, of oleic acid, saponified with 20 parts of sodium silicate in 200 parts of water, is stirred at 100°C. into 5 parts of chlorinated rubber dissolved in 25 parts resin oil; 125 parts of water containing casein 8 and ammonia 0.5 parts is then added.8

Chlorinated Rubber	100.0	Toluol	16.0
Hydrogenated Methyl		Xylo1	110.0
Abietate	15.0	Water	98.5
Oleic Acid	4.9	Caustic Soda 9	0.7

<sup>&</sup>lt;sup>5</sup> Bennett, Chem. Formulary, Vol. 5, p. 72 (1941).

<sup>&</sup>lt;sup>6</sup> Ibid., Vol. 4, p. 98 (1939).

<sup>7</sup> Ibid.

<sup>\*</sup> Ibid., Vol. 3, p. 110 (1936).

<sup>&</sup>lt;sup>9</sup> Billing, U. S. 2,230,792 (1941).

# CHAPTER XVIII

# TEXTILE EMULSIONS

#### TEXTILE SIZINGS

#### FORMULA No. 1.

Batavia Dammar A/D	10	Morpholine	1
Stearic Acid	10	Water	100

Melt the dammar and stearic acid. Keeping the temperature at 90°-95°C., stir in the morpholine and the water which has been preheated to 90°-95°C.

## FORMULA No. 2.

White Beef Tallow	30	Water	32-34
Sulfonated Tallow (50%)	30	Disinfectant or Deodorant	1-2
Japan Wax Emulsion	6-8		

Melt Japan wax and sulfonated tallow while agitating, when thoroughly melted add beef tallow and stir until thoroughly mixed. Then add water gradually and agitate until a full white creamy mix is secured.<sup>2</sup>

#### FORMULA No. 3.

Rosin	24-60	Sodium Silicate	5-10
Linseed Oil	24-60	Water 3	500
Alkaline Casein Dispersion	8-10		

<sup>&</sup>lt;sup>1</sup> Bennett, Chem. Formulary, Vol. 5, p. 591 (1941).

<sup>&</sup>lt;sup>2</sup> P. C.

<sup>&</sup>lt;sup>8</sup> Lefranc, U. S. 1,861,927 (1932).

Water

Paraffin Wax

FORMU	ILA No. 4	
59.6	Oleic Acid	3.0
94	Caustic Soda	3.0

3

Beeswax 11.0 Soluble Olive Oil 4 3.0 Japan Wax Stearic Acid 11.0

#### FORMULA NO. 5.

Linseed Oil	100	Ammonium Linoleate	16
Trichlorethylene	100	Water 5	100-200

## FORMULA NO. 6.

40 Trigamine

Oleic Acid	5 Water	112
•	dissolved in the water and	

is added, stirring thoroughly until completely homogeneous. This is then heated to 65-70°C, and the wax, previously melted, added with rapid agitation. Stirring is continued until the emulsion is cold.6

## FORMULA No. 7.

Sulphonated Castor Oil		Caustic Soda Solution	
(62% T.F.M.)	800	(27° Bé.)	350
Water	550	Silicate of Soda Solution	
		(37° Bé.)	1300

Heat the sulphonated oil to 35-40°C, and slowly add the other ingredients in order given above, maintaining temperature above 35°C. until mixing is completed.

#### FORMULA No. 8.

Melt together at a temperature of 50-60°C, equal parts of olein and dried phosphatides which have a soya bean oil content of 30% (and are obtained by extraction with 90 parts of benzol and 10 parts of 96% alcohol). Then by adding an

<sup>4</sup> Bennett, Chem. Formulary, Vol. 4, p. 529 (1939).

<sup>&</sup>lt;sup>5</sup> Ibid., Vol. 1, p. 475 (1933).

<sup>&</sup>lt;sup>6</sup> Ibid., Vol. 4, p. 553 (1939). <sup>7</sup> Ibid., Vol. 3, p. 365 (1936).

equal quantity of water, keeping the temperature at 50–60°C. and adding small quantities of dilute soda lye, the mixture is emulsified under vigorous stirring. When required for use, this emulsion is diluted with further quantities of water in the desired manner <sup>8</sup>

## FORMULA No. 9.

Rosin	10-15	Stearin	7-20
Olein	2-10	Water	1000
Triethanolamine	7-20		

Sizing is done at 50°C. for 15 minutes. Then raise temperature to 60°C.

Desizing or washing is done with ½% soap solution at 60°C.9 for one hour.

#### FORMULA No. 10.

Sulphonated Castor Oil		Caustic Soda Solution	
(58%)	50 gal.	(27° Bé.)	12 gal.
Paraffin Oil (28° Bé.)	10 gal.	Water	23 gal.
		Steam Distilled Pine Oil	12 gal.

Mix ingredients in order named at 35–40°C., being careful to add caustic soda solution and pine oil very slowly, with constant stirring and allowing mixture to cool to room temperature as the pine oil is being added.<sup>10</sup>

#### ANTI-STATIC TEXTILE DRESSING

Magnesium Chloride	Water	50
(10% Solution)		

Filter and add to the following emulsion previously prepared:

Mineral Oil	80	Oleic Acid	7
Sulphonated Lauryl Alcohol	25	Water	50
Lauryl Alcohol	5		

<sup>9</sup> Bennett, Chem. Formulary, Vol. 2, p. 468 (1935).

<sup>10</sup> Ibid., Vol. 3, p. 365 (1936).

The above is used on cellulose acetate staple fiber to the extent of 1-4%.

#### FUGITIVE TINTING DYE

1. Diglycol Laurate 98 3. Mineral Oil 1000 2. Fugitive Dye 2

Dissolve 2 in 1 and then add 3. Goods marked with the above are washed out easily.<sup>2</sup>

#### KIER PENETRANT OIL

Xylol 10 gal. Sulphonated Castor Oil Water 20 gal. (62% T.F.M.) 20 gal.

Mix in xylol first and then water, with agitation, at 35-

#### LUSTER EMULSION FOR STARCHING

Stearic Acid 1 kg. Water 4 kg. White Beeswax 200 g. Citronella Oil for Perfume 5 g. Borax 60–75 g.

Of this emulsion, 10 spoonfuls are used for 1 kg. of starch.<sup>4</sup>

#### ROT PROOFING EMULSION

1 Zinc Naphthenate	60.0	3 Emulsifying Agent	2.5
2 Mineral Spirits	17.5	4 Water	20.0

1 is dissolved in 2 and added slowly with vigorous mixing to 3 and 4 (mixed) and mixed until emulsified. The emulsion is diluted, as desired, with water before use.<sup>5</sup>

# SCOURING ASSISTANT, TEXTILE

Dupanol L.S. Paste	15	Tetralin	70
Hexalin	2	Water 6	10
Pine Oil	3		

<sup>&</sup>lt;sup>1</sup> Ibid., Vol. 5, p. 595 (1941).

<sup>&</sup>lt;sup>2</sup> Ibid., p. 598.

<sup>&</sup>lt;sup>8</sup> Bennett, Chem. Formulary, Vol. 3, p. 365 (1936).

<sup>4</sup> Ibid., Vol. 5, p. 596 (1941).

<sup>&</sup>lt;sup>5</sup> Ibid., p. 593.

<sup>&</sup>lt;sup>6</sup> E. I. du Pont de Nemours & Co., Wilmington, Del.

## DEGREASING EMULSION

Kerosene Dupanol LS Paste	60 5	Water 7	35
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## SOAPY FULLING OIL

a. Oleic Acid	70	Carbon Tetrachloride	20
Methyl Cyclohexanol	5	b. Caustic Potash (50° Bé.)	

To a add enough b to just neutralize to phenolphthalein.1

## TEXTILE SOFTENERS

		0. 12. 2. 2. 2. 2. 2. 2. 2. 2. 2. 2. 2. 2. 2		
	Formula	No. 1.		
Water	7 3	Tallow 2		
Soap	ა			
	FORMULA	No. 2.		
Tallow	100	Caustic Potash	(45%)3	30
Formula No. 3.				
Lecithin	5 10	Tetralin		500-1000
Cyclohexanol	50–100	Water 4	•	1000
	FORMUL	A No. 4.		
Mineral Oil	10	Dupanol WS		2
Glue, Hide	10	Water 5		<i>7</i> 8

#### TEXTILE GLOSS OIL

## FORMULA No. 1.

Paraffin Wax		20	Water	70
Lanette Wax	sx	10		

Heat together and stir until dispersed.6

<sup>7</sup> Ibid.

<sup>&</sup>lt;sup>1</sup> Bennett, Chem. Formulary, Vol. 4, p. 527 (1939).

<sup>&</sup>lt;sup>2</sup> Ibid., p. 526.

<sup>3</sup> Ibid.

<sup>4</sup> Ibid.

<sup>&</sup>lt;sup>5</sup> E. I. du Pont de Nemours & Co., Wilmington, Del.

<sup>&</sup>lt;sup>6</sup> Bennett, Chem. Formulary, Vol. 2, p. 455 (1935).

## FORMULA No. 2.

Paraffin Wax	20	Glycosterin	10
Petrolatum White	2	Water	180

Heat together and stir until dispersed.7

# FORMULA No. 1.

#### YARN FINISH

	Montan Wax, Bleached, A 10 Water 8 65
--	---------------------------------------

# FORMULA No. 2.

Montan Wax, Double Bleached	8	Potassium Carbonate	2
Paraffin Wax (40/42°C.)	10	Water 8a	60
Rosin	7		

# "SOLUBLE" TEXTILE OILS

Formula 1	No. 1	No. 2
Mineral Oil (55 sec. Saybolt)	70	82
Oleic Acid	15	3
Sodium Petroleum Sulfonates	15	5
Lard Oil	1	10 <sup>2</sup>

#### FORMULA No. 3.

Xylol or Toluol	15 gal.	Caustic Soda Solution	
Paraffin Oil (28° Bé.)	78 gal.	(27° Bé.)	1 gal.
Red Oil	2 gal.	Water	1 gal.
Alcohol	3 gal.		•

Mix the paraffin oil and red oil, heat to 40°C., add the previously mixed water and caustic solution, then add the xylol slowly and the alcohol last and rapidly cooling them as quickly as possible after mixture is uniform.<sup>3</sup>

<sup>7</sup> Ibid.

<sup>8</sup> Ibid., Vol. 5, p. 596 (1941).

<sup>&</sup>lt;sup>8a</sup> Ibid., Vol. 4, p. 188 (1939).

<sup>&</sup>lt;sup>1</sup> Kinney, Can. 284, 807 (1928).

<sup>2</sup> Ibid.

<sup>&</sup>lt;sup>8</sup> Bennett, Chem. Formulary, Vol. 3, p. 365 (1936).

#### FORMULA No. 4.

Water	2.0	Diethylene Glycol	2.0
Caustic Potash	1.2	Oleic Acid	9.9
Light Mineral Oil	85.0		

To the mineral oil add 6 parts of oleic acid, and the caustic potash dissolved in the water. Stir in the diethylene glycol. Then add slowly with constant stirring the remainder of the oleic acid, till the solution becomes clear. If properly made, this oil will emulsify when mixed with water.

#### FORMULA No. 5.

a. Turkey Red Oil (50%)	30	b. Oleic Acid, White	15
Diglycol Laurate	1	c. Caustic Potash To	neutralize
Alcohol	1	Water, To make 5	100
Methyl Glycol	1		

#### FORMULA No. 6.

An oil such as a mineral oil 64, is used in admixture with "Carbitol" 2, corn oil soap 14, rosin 10, water 6 and diethylene glycol 4%.

#### FORMULA No. 7.

a. Oleic Acid Colza Oil	8.0 4.0	b. Alcohol Methyl Glycol	2.0 1.0
Rosin	2.0	c. Caustic Potash (50°)7	3.8
Castor Oil Fatty Acid	2.0		
Spindle Oil	32.0		

#### TEXTILE SCOURING EMULSION

Oleic Acid	10	Trigamine	3
Pine Oil	87	Water	100

Mix 30 grams of the pine oil with the trigamine, and oleic acid and stir thoroughly. Then add slowly 35 grams of water and stir till a smooth thick emulsion is formed.

<sup>4</sup> Ibid., Vol. 4, p. 188 (1939).

<sup>&</sup>lt;sup>5</sup> Ibid.

<sup>6</sup> Ibid., Vol. 3, p. 365 (1936).

<sup>&</sup>lt;sup>7</sup> Bennett, Chem. Formulary, Vol. 4, p. 187 (1939).

Then add the remainder of the oil with high speed agitation and finally the rest of the water.8

#### NON-SLIPPERY BELT AND ROPE PRESERVATIVE

Soap Tallow Beeswax

Melt together and add slowly with vigorous stirring.

Water (Boiling) 30

When emulsion is complete, dilute with water to a density of 0.950.1

# SPINNING OIL FOR LIGHT OR WHITE FIBERS

White Oleic Acid 41 Caustic Potash (48° Bé.) 171/2 Denatured Alcohol 31

Mix these three thoroughly and test for neutrality. Make exactly neutral, then add

Water White Oleic Acid 2 11 35 White Mineral Oil 854

# ROPE PRESERVATIVE

Tallow Water 30 Beeswax 1 Soap 3 3

#### TEXTILE LUBRICANTS

#### FORMULA No. 1.

Olive Oil 87 Trigamine Oleic Acid 10 Water 4 100 cc.

# FORMULA No. 2.

Oleic Acid 40.00 Water 60.00 Triethanolamine 2.04

<sup>8</sup> Ibid., p. 499.

<sup>&</sup>lt;sup>1</sup> Ibid., Vol. 5, p. 593 (1941). <sup>2</sup> Ibid., Vol. 4, p. 188 (1939).

<sup>&</sup>lt;sup>3</sup> Pfahl. Brit., 488, 643 (1938).

Bennett, Chem. Formulary, Vol. 4, p. 187 (1939).

Stir together at about 35°C.,6 and transfer immediately into a homogenizer, where the emulsion is homogenized under about 150 atmospheres pressure.5

#### FORMULA No. 3.

Monoamylamine Oleate Paraffin Oil (28° Bé.)	4 96	Mix the above and stir into Water <sup>6</sup>	100
--	---------	--	-----

#### FORMULA No. 4.

3. Rosin WW	6.4	c. Spindle Oil	73.4
Mineral Oil	20.0	d. Alcohol	5.4
b. Olein, Light	10.5	e. Caustic Soda (40° Bé.)	2.2

Melt up a with agitation, until clear. Add b, c, cool down to 45°C., add d, and, with vigorous mixing, e in small portions, until the cloudiness disappears.

If it does not go clear completely, add some olein with caution.7

#### OIL COATING FOR GLASS FIBERS

Bright Stock (Petro-		Ammonia	(28°)			4	%
leum) Oil		Water 8		$T_0$	make	100	%
Oleic Acid	12.6–18.5%						

#### ACETATE RAYON OIL

Sulphonated Castor Oil		Acetic Acid (28%)	20 gai
(65%)	50 gal.	Paraffin Oil (28° Bé.)	5 gai
Commercial Olive Oil	45 gal.	Water	100 gal

Manipulation: Mix the three oils and the water at 40°C. Then cool to 30°C., and stir acetic acid into mixture slowly.

# WATER PROOFING (ACETATE) RAYON

Paraffin Wax	5 g. Ammonium Hydr	oxide
Soap	5 g. (0.880)	30 cc.
	Water	970 сс.

<sup>&</sup>lt;sup>5</sup> Ibid.

<sup>6</sup> Ibid.

<sup>7</sup> Ibid.

<sup>&</sup>lt;sup>8</sup> Ibid., p. 215.

<sup>&</sup>lt;sup>9</sup> Ibid., Vol. 3, p. 364 (1936).

Heat to 55°C. and stir vigorously until a uniform emulsion results. Soap the rayon in this emulsion at 45°C. for 15 minutes; remove; centrifuge and treat for 10 minutes at 20°C. with a 5°Tw. solution of aluminum acetate.<sup>10</sup>

# RAYON DELUSTERING

Olive Oil Soap (Low Titre) 10 Water White Glue 10

100

Dissolve by warming and stirring; add Paraffin Wax. Heat and stir till emulsified.

Take 21/2 parts of above emulsion and add to it

Water

100

Stir and add

Infusorial Earth 11

2.27-4.54

10 Ibid., Vol. 2, p. 468 (1935).

11 Bennett, Chem. Formulary, Vol. 2, p. 468 (1935).

# CHAPTER XIX

# WATERPROOFING EMULSIONS

# TEXTILE WATERPROOFING EMULSIONS

# FORMULA No. 1.

	Α		
Soap Water Gelatin		Water Paraffin Wax	23.0 20.0
	B		
Aluminum Sulfate Water Soda Ash		Water Formaldehyde	2.0 0.2
Emulsified Mixture A & B Tapioca Water	30.0 7.0 50.0	Titanium Dioxide Water Benzoic Acid <sup>1</sup>	5.0 5.0 2.0
	FORMULA	No. 2.	
Cumar Carnauba Wax Trihydroxyethylamine	4 15	Triethanolamine Water <sup>2</sup>	2½ 185
Stearate	71/2		
	FORMULA	No. 3.	
Ethylene Dichloride Naphtha Diglycol Oleate	40 40 15	Caustic Soda (50%) Glycerin <sup>3</sup>	3 25

<sup>&</sup>lt;sup>1</sup> Scholler, H. S., 2, 230, 656 (1941).

<sup>&</sup>lt;sup>2</sup> P.C.

<sup>8</sup> P.C.

# · Practical Emulsions

	FORMULA	No. 4.	
Ethyl Cellulose Hydrogenated Methyl Abietate Toluol Butanol	16.6 3.4 56.0 16.0	Naphtha Water Sodium Lauryl Sulfate Sulfonated Castor Oil 4	8.0 1500.0 7.5 15.0
	FORMULA	No. 5.	
Japan Wax Paraffin Wax Duponol WA Paste	80.6 9.0 5.7	Caustic Soda (10% sol.) Perfume 5 To	5.7 suit
	FORMULA	No. 6.	
Japan Wax Tallow Oil Diglycol Laurate Water	17½ 17½ 15 50	Mineral Oil Carbitol Diglycol Laurate Water <sup>6</sup>	40 3 12 45
	FORMULA	No. 7.	
Paraffin Wax Carnauba Wax Triethanolamine Linoleate	2 2 10	Borax Water <sup>7</sup>	3 30
	FORMULA	No. 8.	
Paraffin Wax Mineral Oil	10 50	Ammonium Stearate (Paste) Water 8	20 70
	Formula	No. 8a.	
Hard Paraffin Wax Petrolatum, Yellow Peanut Oil Beeswax, Yellow	50 400 200 100	Lanolin, Anhydrous Rosin, Pale Water Borax <sup>8a</sup>	60 40 140 10
	FORMULA	No. 8b.	
Paraffin Wax Resin RH 35 Gum Arabic	15 15 5	Water Preservative (Moldex) <sup>8b</sup>	70 ½10

<sup>&</sup>lt;sup>4</sup> Billing, U. S., 2, 230, 792 (1941). <sup>5</sup> E. I. du Pont de Nemours & Co., Wilmington, Del.

<sup>6</sup> P.C.

<sup>7</sup> P.C.

<sup>8</sup> P.C.

<sup>8</sup>a P.C.

sb P.C.

#### WATERPROOFING FOR LEATHER

Gasoline	100 cc.	Rubber Latex	100 cc.
Paraffin Wax	25 g.	Soap (10% Solution) <sup>9</sup>	50 cc.
Paraffin Oil	10 g.	•	

# STUCCO WATERPROOFING

Sodium Stearate 5 lb. Water 95 lb.

Warm to 50°C. and stir till uniform, then add

Suet 2 lb. Cresol Emulsion 10

<sup>&</sup>lt;sup>9</sup> Bradley, U. S. 1,860,651 (1932).

<sup>&</sup>lt;sup>10</sup> Bennett, Chem. Formulary, Vol. 3, p. 239 (1936).

# CHAPTER XX

# WAX EMULSIONS

#### BENTONITE WAX EMULSIONS

Make a 5% suspension of bentonite, that is, 95% water and 5% bentonite. The easiest way to do this is to put all the water to be used in a vessel and sprinkle a little of the bentonite on the surface. Allow to stand about three minutes or until the bentonite sinks to the bottom. Repeat. Don't stir or agitate. Allow to stand for at least four hours, then stir or agitate.

Heat the wax to fluidity. Heat the bentonite dispersion to the same temperature or slightly higher. Pour the wax into the water and stir. Stirring should be particularly vigorous at the point where the wax begins to solidify. A mechanical agitator is the best. In the laboratory use an electric soda-fountain stirrer.

The final mixture may be between 20% to 50% wax. However, it is best not to use over 40% wax, otherwise the emulsion will be too viscous.

The pH of bentonite in water is 7.5–7.8. In most emulsions a slightly higher pH is desirable, for instance, about 8.5. It would be well to adjust the water dispersion to the higher pH by very slight additions of alkali. Calcium oxide, calcium hydroxide, caustic calcined magnesite, magnesium oxide are recommended.

In some cases it will be found that a thicker dispersion of bentonite and water is desirable. This can be accomplished by using 6% bentonite or by adding very small percentages of other ingredients to the 5% dispersion. These other ingredients should be added after the dispersion is made. They will combine the advantages of thickening the dispersion and at the same time raising the pH. Add minute quantities and stir, using just enough to thicken the gel.

In relation to the quantity of bentonite in the dispersion, proper proportions would be as follows:

Calcium Oxide	3%	Caustic Calcined	
Calcium Hydroxide	3%	Magnesite	½ to 1%
Chloride of Lime	2%	Magnesium Oxide	½ to 1%

For instance, with 95 cubic centimeters of water and 5 grams of bentonite, you would use 15/100 of a gram of calcium oxide, that is 150 milligrams. The magnesia compounds react more slowly. The others mentioned, react practically immediately.<sup>1</sup>

#### CANDELILLA WAX EMULSIONS

a. Candelilla Wax	30	b. Borax	21/2
Triethanolamine	7	Water	20
Red Oil	5	c. Water 2	150

#### FORMULA No. 2.

a. Candelilla Wax 20 Oleic Acid

Heat to 95–100°C. and then run in the following heated to 95–100°C., mixing with a high-speed stirrer:

b. Triethanolamine Water : 185

#### FORMULA No. 3.

a. Candelilla Wax 20 Oleic Acid

<sup>&</sup>lt;sup>1</sup> Bennett, Chem. Formulary, Vol. 2, p. 182 (1933).

<sup>&</sup>lt;sup>2</sup> Ibid., Vol. 4, p. 96 (1939).

<sup>8</sup> P. C.

h Triathanalamina

# Use above method adding:

Borax	11/2	
14	FORMULA No. 4.	
Candelilla Wax Ammonium Linoleate S	23 Water 3 Ammonia	232 2

Water 88

185

Melt wax at 100°C. The ammonium linoleate is thinned by working in 30 cc. of water, a little at a time. The balance of water is heated to 95–100°C., and the ammonium linoleate solution is added to it. The whole is boiled and added, a little at a time, while stirring vigorously. Finally add the ammonia and stir until cooled to 50°C.

# CARNAUBA WAX EMULSIONS FORMULA No. 1.

Carnauba Wax Trihydroxyethylamine	260.00	Borax Caustic Soda (50%)	11.11 2.2 <b>2</b>
Oleate	48.50	Water <sup>5</sup>	650.22
Triethanolamine	28.00		
	Formul	A No. 2.	
Carnauba Wax	16.0 g.	Ammonium Linoleate	2.4 g.
Kerosene	20.0 cc	Water	200.0 cc.

The ammonium linoleate is placed in a vessel and covered with the water (cold) and allowed to stand overnight. The following day it is warmed and stirred until completely dispersed in the water, taking care that no lumps are left. This is taken to 90°C. and stirred by means of a high speed mixer. The wax is melted, taken to 100°C., and the kerosene added and stirred until the wax has dissolved in it. This is then added to the hot ammonium linoleate dis-

5 Ibid.

<sup>8</sup>a P. C.

Bennett, Chem. Formulary, Vol. 5, p. 72 (1941).

persion and the agitation continued until the emulsion is cool. This gives a fluid emulsion.

FORMULA NO	A No. 3.
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Mineral Oil (Spindle) 19 cc. Ammonium Linoleate 2.4 g. Carnauba Wax 18 g: Water 7 102 cc.

FORMULA No. 4.

Hydromalin 138 Carnauba Wax No. 1 or No. 2 Yellow 250

Heat to 120°C.-140°C, half an hour.

Cool to 100°C.-105°C.

Add to the above slowly with stirring.

Water (at 100°C.) 280

Stir to smooth paste then add slowly with good stirring.

Water (at 100°C.) 1500

Keep as close to 100° as possible for 15 minutes with rapid stirring. Then stir till cool.8

#### FORMULA No. 5.

a. Carnauba Wax	90 g.	b. Caustic Soda (50%)	б д.
Oleic Acid	10 g.	Water	24 g.
Trigamine	10 g.		

a is melted. b is mixed together and heated. It is added to a slowly with stirring and the whole stirred until it begins to solidify. One part of the above disperses easily in five parts of boiling water.<sup>9</sup>

#### FORMULA No. 6.

Carnauba Wax	20.0	Borax	1.0
Duponol WA Paste	8.0	Water	71.0

<sup>&</sup>lt;sup>6</sup> Bennett, Chem. Formulary, Vol. 1, p. 162 (1933).

<sup>7</sup> Ibid.

<sup>&</sup>lt;sup>8</sup> Glyco Products Co., Inc., Brooklyn, N. Y. (Catalog).

<sup>9</sup> Ibid., Vol. 4, p. 475 (1939).

Melt the carnauba wax in a steam bath and pour it into the Duponol WA Paste and borax dissolved in the water at approximately 95°C. Stir vigorously until a smooth emulsion is formed. Then stir gently until the emulsion has cooled to room temperature. A variation of this procedure is to add the Duponol WA Paste to the wax which has been melted in a steam bath and then to add the borax, dissolved in the water. Do not stir vigorously until cool.<sup>10</sup>

## FORMULA No. 7.

Carnauba Wax	30.0	Gelatin 0.30	to 1.3
Oleic Acid	3.3	Shellac Solution consists of	
Caustic Soda	0.5	Refined Wax Free Shellac	1.5
Triethanolamine	2.7	Borax	0.38
Shellac Solution	62.0	Water 11	8.33
Water	260.0		

# CARNAUBA WAX-RESIN EMULSIONS

FORMULA						
	No. 1	No. 2	No. 3	No. 4	No. 5	
Carnauba Wax No. 1	45	45	30	30	30	
Gum Dammar A		15	30	30	30	
Manila Gum DBB		5				
Caustic Soda	1	1.5	0.9	2	2	
Water	420	420	422	422	422	
Triethanolamine	4	4				
Trigamine			9.9	9.5	19	
Oleic Acid	9	9	7	7	14	
Stearic Acid						
"Run" Boea (Hard Bold Amber)						
"Run" Congo						
	Film-	Film	Film	Film	Film	
	Dull	Same	Same	Same	Same	
	Slight	as	as	as	as	
F	Rubbing	No. 1	No. 1	No. 1	No. 1	
Gi	ves Good					
	Gloss					

<sup>10</sup> E. I. du Pont de Nemours & Co., Wilmington, Del.

<sup>11</sup> Steinle, U. S. 2,275,659 (1942)

T. T.	ORMULA				
	No. 6	No. 7	No. 8	No. 9	No. 10
Carnauba Wax No. 1	33.5	47	47	30	30
Gum Dammar A	26.5	13	13		
Manila Gum DBB					
Caustic Soda	1.5	0.6	0.6	1	0.5
Water	. 420	420	420	420	420
Triethanolamine	. 2	4	4	4	4
Trigamine					
Oleic Acid		• • •	9	9	9
Stearic Acid		9			
"Run" Boea (Hard Bold Amber)	)			30	
"Run" Congo					
3	Film	Film	Film	Film	Film
	Glossy	Glossy	Glossy	Glossy	Glossy

The procedure used is to melt the wax and to incorporate in it the natural resin (at temperatures of 100°-150°C.). This mixture is permitted to solidify and then used as needed in making the emulsion. For this purpose, the resin-wax mixture is melted in a boiling water bath. The emulsifier is added, the oleic acid being first incorporated, followed by the triethanolamine or trigamine. The sodium hydroxide is dissolved in 25 parts of the water, the solution heated, and added at its boiling point. This sometimes causes a gel to form, which is the signal to add the remainder of the water at its boiling point. If a gel does not form, addition of the water is withheld for a few minutes to permit thorough mixing of all constituents. Nos. 1 and 2 are made slightly different in that the Manila resin is dissolved in the sodium hydroxide solution mentioned above and not in the wax.<sup>10a</sup>

#### CERESIN WAX EMULSION

1.	Ceresin Wax	16	3.	Water	80
2	Aminostearin	4			

Heat 1 and 2 to 85°C., and add slowly with vigorous mixing to the water at 90–100C.<sup>11a</sup>

<sup>&</sup>lt;sup>10a</sup> Amer. Gum Importers' Ass'n. <sup>11a</sup> Ibid., Vol. 5, p. 72 (1941).

#### ESPARTO WAX EMULSION

a. Esparto Wax	15	b. Borax	11/4
Triethanolamine	3	Water	10
Red Oil	17/8	c. Water	321/2

Heat a to 95–100°C, and mix well; run into b heated to 95-100°C, beating vigorously; when smooth gel is formed add c heated to 95-100°C. and beat vigorously until cool.12

#### HALOWAX EMULSIONS

	FORMUL	A No. 1.	
Water Halowax		Stearic Acid Triethanolamine 18	53 g. 27 g.
IIalowax			21 g.
	FORMUL	A. No. 2.	
Water	3 lb.	Stearic Acid	108 g.
Halowax	3 lb.	Triethanolamine 14	54 g.
	FORMUL	A No. 3.	
Water	3 lb.	Stearic Acid	108 g.
Halowax	13 oz.	Triethanolamine	54 g.
Halowax Oil No. 1000	3 07.		

Melt the wax and stearic acid together and stir. Heat the water and triethanolamine until they start to boil. Add the wax to the water and stir with an electric stirrer and then run through the colloid mill. A good emulsion is obtained if it is cooled quickly after coming from the colloid mill.15

# JAPAN WAX ("SOLUBLE")

a. Japan Wax	50	b.	Trigamine
Stearic Acid	14		_

 <sup>12</sup> Ibid., Vol. 4, p. 96 (1939).
 18 Bennett, Chem. Formulary, Vol. 1, p. 162 (1933).

<sup>14</sup> Ibid. 15 Ibid.

An emulsion is obtained by dispersing one part of above in eight parts of hot water.<sup>18</sup>

# JAPAN WAX EMULSION

a. Japan Wax
Trihydroxyethylamine
Stearate

36
b. Water, Boiling
360
4

Warm a to 65°C, and add b to it slowly with vigorous stirring.<sup>17</sup>

#### SELF-EMULSIFYING LAUNDRY WAX

Japan Wax	80.6	Caustic Soda (10% Solution)	5.7
Paraffin Wax	9.0	Perfume	q.s.
Duponol WA Paste	4.7		

Mix the waxes, DUPONOL WA Paste, and caustic soda solution together in a steam jacketed vessel and stir until thoroughly homogeneous. Cease heating and stir occasionally until almost solid. Then pour into suitable molds to solidify.<sup>18</sup>

#### MONTAN WAX EMULSIONS

#### FORMULA No. 1.

Montan Wax 20 Oleic Acid 5

Heat to 100°C. and add the following heated to 95-100°C slowly with good mixing:

Triethanolamine	Water 1	185
Borax		

#### FORMULA No. 2.

Montan Wax	60 g.	Bentonite (6.6%	5 cc.
Creosote	40 cc.	Dispersion) <sup>2</sup>	
Water	100 cc.	• •	

<sup>16</sup> Ibid., Vol. 4, p. 475 (1939).

<sup>&</sup>lt;sup>17</sup> Ibid., p. 96.

<sup>18</sup> E. I. du Pont de Nemours & Co., Wilmington, Del.

<sup>&</sup>lt;sup>1</sup> Bennett, Chem. Formulary, Vol. 4, p. 96 (1939).

<sup>2</sup> P. C.

#### FORMULA No. 3.

Bleached montan wax is fused together with an equal amount of a solid, wax-like polymerization product of ethylene oxide (softening point about 50°C.) obtained by allowing ethylene oxide to stand at about 10° below zero C. with about 1 per cent its weight of sodium oxide or by heating ethylene oxide with about 0.1 per cent of caustic soda solution to from 50° to 60°C., the melt then being heated in ten times its weight of water while stirring. An emulsion is obtained which after cooling becomes very viscous and which, if desired, may be further diluted with water while heating, without deflocculation of the wax, and may be employed as a polish. Instead of Montan wax, stearic acid, montanic acid, tallow, wool fat and similar substances may be emulsified in an analogous manner.<sup>3</sup>

# FORMULA No. 4.

Montan Wax	160	Water	600
Ammonium Linoleate	40		

Dissolve the ammonium linoleate in the water and heat to 95–100°C.; pour the wax into this slowly while stirring vigorously. While still hot run through a colloid mill.<sup>4</sup>

#### "OPAL WAX" EMULSION

Add desired weight of wax to weighed portion of water (emulsions having 40% wax, 60% water form a thick cream. Higher proportions of wax will be solid on cooling). Heat the water to boiling and continue boiling until wax has completely melted and formed a homogeneous oil film on the surface of the water. Remove from hot plate and after boiling has ceased add NH<sub>4</sub>OH while stirring until the melt has a slight but definite ammoniacal odor. Place

4 Ibid., p. 182.

<sup>&</sup>lt;sup>3</sup> Bennett, Chem. Formulary, Vol. 2, p. 175 (1935).

in a mechanical agitator which will give rapid and thorough stirring (e.g., for small lots the type of mixer used at soda fountains is entirely satisfactory) and continue this stirring until emulsion has cooled below 60°C. Slow cooling in the range from 85° to 70°C. is advantageous.

Any froth or foam which forms will break upon standing except in the case of the higher wax contents (30% and above), when it may be necessary to apply vacuum to break the foam completely.

In the case of the higher wax contents, only very slow cooling while stirring will prevent the formation of a few small granules of solid wax.<sup>5</sup>

#### FORMULA No. 2.

a. Opal Wax	15	b. Water	75
Aminostearin	5	Sulfatate (Wetting Agent)	5

Heat a to 100–105°C. and add to it slowly while mixing with high-speed mixer b heated to 95°C.

#### OURICOURI WAX EMULSION

Ouricouri Wax	13.2	Borax	1.0
Diglycol Oleate	2.5	Water	80.0
Triethanolamine	2.2		

Melt wax and diglycol oleate at 80°C., and make solution of other ingredients at same temperature. Add latter to former slowly while mixing vigorously with electric stirrer.<sup>7</sup>

#### PARAFFIN WAX EMULSIONS

#### FORMULA No. 1.

a. Paraffin Wax	c. Trigamine	3
b. Stearic Acid	d. Water	80

<sup>&</sup>lt;sup>5</sup> Bennett, Chem. Formulary, Vol. 2, p. 189 (1935).

<sup>6</sup> Ibid., Vol. 4, p. 96 (1939).

<sup>7</sup> Ibid., Vol. 5, p. 72 (1941).

Melt a and b at 65–70°C. and add c and d at same temperature, slowly, while mixing with a high-speed agitator.

FORMULA No. 2.

Paraffin Wax

120 g. Stearic Acid

12 g.

Melt together and while stirring vigorously add following heated to 55°C.

Ammonia (26° Bé.)

6 cc. Water

182 cc.

Stir until uniform.9

FORMULA No. 3.

Glyceryl Monostearate

5 Water

150

Heat and stir vigorously until uniform. Pour into this slowly while stirring strongly:

Paraffin Wax (melted) 10

40

FORMULA No. 4.

Paraffin Wax

25 Glycol Stearate

5

Melt together and while stirring vigorously add

Water (boiling) 11

175

#### FORMULA No. 5.

Mix 33 parts of paraffin wax with 3 parts of oleine, and pour this mixture into a solution of 0.6 part of strong ammonia in 63.4 parts of water, heated to 160°F.<sup>12</sup>

# PARAFFIN WAX ("SOLUBLE")

FORMULA No. 1.

a. Paraffin Wax Stearic Acid 50 b. Trigamine

7

8 Ibid., Vol. 4, p. 96 (1939).

<sup>&</sup>lt;sup>9</sup> Bennett, Chem. Formulary, Vol. 3, p. 108 (1936).

<sup>10</sup> Ibid.

<sup>11</sup> Thid.

<sup>12</sup> Ibid.

Melt a to about 70°C. Turn off the heat and add b stirring thoroughly. Continue stirring till it begins to solidify. One part of the above disperses easily in two parts of hot water.13

#### FORMULA No. 2.

Paraffin Wax

77 Duponol WS 14

23

Ibid., Vol. 4, p. 475 (1939).
 E. I. du Pont de Nemours & Co., Wilmington, Del.

# CHAPTER XXI

# MISCELLANEOUS

# NON-AQUEOUS EMULSION

Wool Grease	20	Methyl Acetone	50
Benzol	50	Alcohol <sup>1</sup>	<b>25</b>
Balata Solution (1 lb. to 1 gal.)	50		

#### HIGH TEMPERATURE STABLE NON-AQUEOUS EMULSIONS

FORMULA	No. 1	No. 2	FORMULA	No. 1	No. 2
Mineral Oil	40	40	Polyglycerol Soya		
Calcium Glyceryl			Bean Ester		5
Stearate	5		Glycerin	60	60

Heat to 200°C. and keep stirring while heating.2

# SAFETY-PAPER INK

An emulsion of a suitable printing ink consistency comprises Canadian balsam 8–20, turpentine 5–17, finely divided substantially colorless mineral matter such as diatomaceous silica 8–25 and castor oil 12–30%, emulsified with a slightly alkaline solution of borax and contains a substantially colorless indicator such as phenolphthalein reactive to both acids and alkalies, so that it is suitable for "safety paper" for checks, etc.<sup>3</sup>

<sup>&</sup>lt;sup>1</sup> Simon, U. S. 1,657,323 (1928).

<sup>&</sup>lt;sup>2</sup> P. C.

<sup>&</sup>lt;sup>3</sup> Bennett, Chem. Formulary, Vol. 4, p. 147 (1939).

#### NON-FOAMING OIL EMULSION

Stearic Acid	Miner	ral Oil	500 g.
Oil-Soluble Mahogany	Water	r	5000 cc.
Sulphonate	99 g.		

Warm and mix first three ingredients until clear; then add water slowly with vigorous mixing.4

#### PETROLEUM DEMULSIFIERS

# FORMULA No. 1. Diglycol Laurate 83 Phenol 4 Sodium Silicate 5 Water 1½ Rosin Soap 5 Paraffin Wax 1 1½

#### FORMULA NO. 2.

5% of a solution of the following is agitated with the emulsion and then allowed to settle:

Naphthenic Acid	¼ Water ²	991/2
Salt		

# FORMULA No. 3.

Furfuraldehyde-Potassium		Ammonia (0.897)	8.9
Bisulphite Solution	11.1	Castor Oil 3	29.6
Ricinoleic Acid	50.4		

#### FORMULA No. 4.

The oil is mixed in continuous phase with

Caustic Soda	25	Glycerin	10
Sodium Acetate	10	Water 4	35–45
Alcohol	20		

<sup>4</sup> U. S. 2.052.164.

<sup>&</sup>lt;sup>1</sup> Bennett, Chem. Formulary, Vol. 3, p. 114 (1936).

<sup>&</sup>lt;sup>2</sup> Ibid., Vol. 4, p. 100 (1939).

<sup>&</sup>lt;sup>3</sup> Ibid., p. 99.

<sup>4</sup> Ibid.

# CHAPTER XXII

# DISPERSIONS

# DISPERSING INSOLUBLE POWDERS

Insoluble powders such as zinc oxide, sulphur, Neozone D, Zenite, dry powder colors and fillers are dispersed by grinding in water with a protective colloid and a dispersing agent. The presence of a small amount of fixed alkali in such a mixture is desirable.

Insoluble Powder	100.00	Sodium Hydroxide	0.10
Casein	3.00	Water	95.65
Preservative "Moldex"	0.25		
"Darvan" (Dispersing			
Agent)	1.00		

Heat the water to 175°F. and add 1 part of 29% aqua ammonia. Add slowly with rapid stirring 3 parts of casein and continue stirring until it is dissolved. Then add 0.1 part of sodium hydroxide. Next 0.25% of a preservative should be added. Darvan (1.0 part) is readily soluble in water and may be added to the solution alone or with the insoluble powders, 100 parts of which are next added. The mixture is then placed in a pebble mill having a capacity double the volume of the mixture and ground for about 24 hours or ground in a colloid mill for a suitable length of time. If dry color pigments are being dispersed, it is desirable to double the grinding time.

<sup>&</sup>lt;sup>1</sup> Bennett, Chem. Formulary, Vol. 5, p. 73 (1941).

#### CARBON BLACK DISPERSION

Carbon Black "Darvan" 100 Water 4 to 8

563 to 599

If a thicker dispersion than results from the above is desired, it may be obtained by adding during the last hour of grinding 1 to 2 parts of casein in water solution.

The method used is similar to that for suspending insoluble powders.<sup>2</sup>

#### CARBON BLACK DISPERSANT

Quebracho Powder (Finely Ground) Soda Ash 95.0 Phenol 5.0 0.2

Mix and grind in a pebble mill.3

#### DISPERSING AGENTS

FORMULA No. 1.

Tallow

175 Dimethyl Amine (25%)

324

Heat at 200°C. in an autoclave for 16 hours. This yields a mixture of dimethyl amides.<sup>4</sup>

#### FORMULA No. 2.

Lauryl Alcohol, Melted

186 Chlorsulphonic Acid

116

Stir while heating gently.

After removing the hydrogen chloride by passing through the reaction mixture current of inert gas, add

Triethanolamine 5

150

#### FORMULA No. 3.

325 kilograms of octadecenylamine are sulphonated at 25°C. with 400 kilograms of concentrated sulphuric acid. After the product has become water-soluble, it is pressed

<sup>&</sup>lt;sup>2</sup> Ibid.

<sup>3</sup> Ibid.

<sup>4</sup> Bennett, Chem. Formulary, Vol. 4, p. 99 (1939).

<sup>5</sup> Ibid.

on to ice, washed with saturated Glauber's salt solution in order to remove the excess of sulphuric acid and finally neutralized with caustic soda solution.<sup>6</sup>

#### FORMULA No. 4.

			20
Sulfonated Castor Oil	35	Water	30
Steam Distilled Pine Oil	35		

Heat the castor or red oil, agitate while adding the pine oil until thoroughly blended, add water—then adding a 25% solution of caustic soda solution with stirring until the solution becomes clear. Test 10 cc. in 50 or 100 cc. of cold water should dissolve instantly and no separation should occur.<sup>7</sup>

#### FORMULA No. 5.

Water	50 Sulf. Castor Oil (75%)	r
Caustic Soda	16 Red Oil	6- 8
	Cresylic Acid	25-32

Add caustic to water then castor or red oil is added while being stirred until solution clears. Cresylic is best added before oil.<sup>8</sup>

#### FORMULA No. 6.

Octyl alcohol is used in the following proportions by volume:

- (a) Pigments in water or oil base paints, use 0.02-1.0% of weight of pigment.
- (b) Pigments in water base cosmetic lotions, use 0.001-0.01%.
- (c) Pottery casting clays to increase "slip" and remove air bubbles, use, 0.0001-0.001% of "slip".
- (d) Leather dressings, spray 0.002– 0.02% onto pigment before water is added.
- (e) Pigments in lacquers, use 0.1-1.0% of weight of pigment.
- (f) Mercerizing baths.
- (g) Penetrating oils, use approximately 1.0%.9

<sup>&</sup>lt;sup>6</sup> Ibid., Vol. 2, p. 184 (1935).

<sup>7</sup> P. C.

<sup>8</sup> P. C.

<sup>9</sup> P. C.

# INDEX

Abrasive polish, 412 Acetate rayon oil, 431 Acid creams, 269 Acriflavine emulsion, 281 Agar, 11 Agitation, 3 Agitation, optimal speed of, 43 Agricultural spray emulsions, formulas of, 203 Agricultural sprays, 82 Albumen, 24, 35 Alginates, 1 Alkalies, 12 All purpose creams, 251 Almond cream, liquid, 271 Almond oil emulsion, 257 Alphabetical list of emulsifying agents, 114 Alphabetical list of surface-active agents, 114 Alum cream, 270 Aluminum and other metals, protective coating for, 405 Anion surface active compounds, 105 Anti-foaming agent, radiator, 296 Anti-knock fuel, 311 Antiseptic cream, 281 Antiseptic emulsion, 292 Antiseptic liquid, 338 Antiseptic oil, water-dispersible, 338 -Anti-static textile dressing, 425

Arabates of sodium, magnesium and iron, 4
Arabic acid, 4
Artificial cream, 298
Artificial latex, 421
Artificial milk, 1
Asphalt emulsions, 83
Asphalt emulsions, formulas of, 232
Asphalt and tar emulsions, 42
Astringent creams, 270
Automobile polishes, 354
Auto radiator cleaner, 243
Avocado oil cleansing cream, 271
Avocado tissue cream, 271

B

Bartlett-Snow colloid mill, 49 Bath milk, 256 Bath oil, 257 Beauty milk, 256 Belt and rope preservative, nonslippery, 430 "Bend" colored bottom finish, 389 Bentonite, 17, 39, 40, 83, 84 Bentonite wax emulsions, 436 Benzene and sodium oleate emulsion, 20 Bile, 9, 10 Bile salt, 10 Bitter almond oil emulsion, 302 Bituminous emulsions, 1, 2, 86 Bituminous emulsions, formulas of, 232 Black bottom gloss, 386

454 Index

Black pigmented lacquer emulsion, Bleaching cream, 272 Borax and fatty acid, 10 Boro-glycerin lanolin cream, 272 Bottom cream, brown, 391 Bottom cream, white, 391 Bottom finish, "bend" colored, 389 Bottom finish, brown, 389 Bottom finish, "natural," 387 Bottom finish, pale pink, 387 Bottom gloss, black, 386 Bottom gloss, brown, 386 Bottom gloss, white, 391, 397 Bottoms, heels and edges, combined ink for, 392 Bottom stain, "natural leather," 388 Breaking emulsions, 77 Breaking of mayonnaise, 96 Breaking of paint emulsions, 99 Bright-drying (rubless) floor polish emulsions, 366 Brown bottom cream, 391 Brown bottom finish, 389 Brown bottom gloss, 386 Brown russet stain, 390 Burnishing ink, 396 Butterscotch icing, 297

Calcium caseinate, 17 Calcium gallate, 4 Calcium gluconate, 4 Calcium lignin sulfonate, 13 Calcium salicylate, 4 Calcium xylene sulfonate, 13 Candelilla wax emulsions, 437 Carbon black dispersant, 451 Carbon black dispersion, 451 Carbon, finely divided, 17 Carnauba wax emulsions, 438 Carnauba wax-resin emulsions, 440 Casein, 1, 24 Castile soap, 17 Castor oil emulsion, 336 Cation active compounds, 105 Causes of instability, 71

Cellulose ester emulsions, 418 Ceresin wax emulsion, 441 Cheese, ice-cream and salad stabilizer, 299 Chemical activity, 80 Chemical breaking of emulsions, 77 Chlorinated rubber emulsions, 422 Chocolate milk, stabilized, 297 Cholestandiol, 5 β-Cholestanol, 5 Cholestanolon, 5 Cholesterol, 5 Cholesterol-lecithin cream, 272 Chondrus, 11 Chromium plate cleaners, 244 Cleaner, auto radiator, 243 Cleaner emulsion for printing rolls, 249 Cleaner, graphite, 244 Cleaner, greasy leather, 245 Cleaner, ink, 245 Cleaner, leather, 245 Cleaner, metal, 246 Cleaner, painted woodwork, 246 Cleaners, chromium plate, 244 Cleaners, formulas of, 239 Cleaner, soap paste paint, 246 Cleaners, rug, 248 Cleaner for wooden surfaces, 249 Cleanser for rubber rugs, 250 Cleanser, upholstery, 248 Cleansing cream, avocado oil, 271 Cleansing creams, 257 Cocoa icing, 297 Colchicine emulsion, 339 Cold creams, 7, 8, 260 "Cold roller grease briquets," 326 Colloidal solutions, viscosity of, 60 Colloid mill, requirements of, 49 Colloid mills, 47 Colloids, natural, 1 Colored emulsions, 102 Combined ink for bottoms, heels and edges, 392 Coprosterol, 5 Cosmetic emulsions, 89, 251 Cosmetics, emulsifying agents used in, 90 Cream, almond, liquid, 271

Cream, alum, 270 Cream, antiseptic, 281 Cream, artificial, 298 Cream, avocado oil cleansing, 271 Cream, avocado tissue, 271 Cream, bleaching, 272 Cream, boro-glycerin lanolin, 272 Cream, cholesterol-lecithin, 272 "Cream" for confectionery, synthetic, 298 Cream formation, speed of, 62 Cream, honey and almond type, 269 Cream, hormone, 273 Creaming, 62 Creaming, retardation of, 62 Cream, lemon juice, 273 Cream, lime and glycerin, 274 Cream, massage, 280 Cream, mosquito repelling, 280 Cream, nail, 280 Cream, peroxide, 272 Creams, acid, 269 Creams, all purpose, 251 Creams, astringent, 270 Creams, cleansing, 257 Creams, cold, 260 Creams, errors in making, 90 Creams, frost bite prevention, 331 Creams, hair, 283 Creams, nourishing, 275 Creams, perspiration deodorizing, 267 Creams, protective hand, 279 Creams, shaving, 288 Cream, sports, 280 "Cumar" emulsion, 416 Cutting oils, 92. Cutting oils, formulas of, 216

D

Dairy product and stabilizer, 299
Dark brown heel and edge stain, 388
Decomposition, 71
Defoaming agents, 294
Defoaming agents, list of, 197
Defrothing agents, 295
Degreasing emulsion, 427

De-inking printed paper, 353 Delustering, rayon, 432 Demulsifiers, petroleum, 449 Demulsifying agents, list of, 197 Deodorizing creams, perspiration, 267 Detergent emulsions, 93 Dextrin, 9 Diethylene glycol esters, 28 Difference of potential between the phases, 76 Digitonin, 6 Dihydrocholesterol, 5 Dihydroxystearates of magnesium and zinc, 4 Disinfectant emulsions, 289, 339 Disinfectant, pine oil, 291 Dispersing agents, 103, 451 Dispersing agents, commercial, 103 Dispersing insoluble powders, 450 Dispersion, excessive, 71 Dispersion, insufficient, 71 Dispersions, 450 Draves-Clarkson test, 105 Drug emulsions, 251 Dry cleaners' soaps, 239 Dual emulsions, 14, 15, 16

E

Edge filler for shoe factory use, 324 Edge ink, one setting, 392, 393, 395 Effect of emulsification on chemical reactions, 80, 81 Egg albumin, 9 Egg-white foam, 34 Egg yolk, 2, 10, 11 Electrolytes, 72 Emulsification by ultrasonic waves, Emulsifier interface, 69 Emulsifiers, classification of, 3, 10 Emulsifiers, mixed, 10 Emulsifying agent, improper, 73 Emulsifying agents, 1 Emulsifying agents, formulas of, 227 Emulsifying agents, insoluble, 29 Emulsifying agents, list of, 114

Emulsifying agents, references for, 131
Emulsifying equipment, 42
Emulsifying high melting materials, 41
Emulsifying power, 3, 4
Emulsion for high-lustre paper, 352
Emulsions, list of, 145
Emulsion solvent soaps, 241
Emulsions, quick breaking, 68
Emulsions, references for, 176
Equipment, emulsifying, 42
Esparto wax emulsion, 442
Ethanolamine soaps, 4
Ethyl cellulose emulsions, 416
Ethylene glycol esters, 28
Excessive dispersion, 71

F

Face creams, 90 Factors determining the type of dispersion, 2, 3 Fat liquors, 320 Fat solvent emulsions, 1 Fatty acid soaps, 9 Fatty alcohol and solvent combinations, 6 Fatty sulfonates, 9 Floor polish emulsions, bright-dryir.g, (rubless), 366 Floor polishes, 364 Foamapin, 35 Foam-breakers, 32 "Foamex," 294 Foams, 31 Food emulsions, 94, 297 Formulas of agricultural spray emulsions, 203 Formulas of asphalt emulsions, 232 Formulas of bituminous emulsions, 232 Formulas of cleaners, 239 Formulas of cutting oils, 216 Formulas of emulsifying agents,

Formulas of miscible oils, 216

Formulas of soluble oils, 216

Formulas of soaps, 239

Formulation, 41
Freckle preventative, 269
Frost bite prevention creams, 331
Frothing and protective action, 12
Fruit coating wax, 214
Fruit protective emulsions, 214
Fuel, anti-knock, 311
Fugitive tinting dye, 426
Fulling oil, soapy, 427
Fur glaze, 411
Furniture polishes, 371

G

Gasoline emulsions, 308 Gasoline, solidified, 311 Gelatin, 1, 11, 17, 22 Gelva resin emulsion, 416 General ink, 393 Glass fibers, oil coating for, 431 Glass polish, 412 Glaze, fur, 411 Gloss ink, 396 Gloss oil, textile, 427 Gloss, white, 397 Gloss, white bottom, 397 Glyceryl esters, 27, 28 Glyceryl ester of stearic acid, 27 Glycols, 9 Graphite cleaner, 244 Graphite lubricant, 327 Gray pigmented lacquer emulsion, 319 "Grease briquets, cold roller," 326 Grease, horse-shoe, 326 Grease, lubricant, 325 Grease, mixed-base, 326 Greasy leather cleaner, 245 Green sulfonates, water-soluble, 8 Gum acacia, 10, 11, 39 Gum arabic, 9, 21 Gums, 24, 39, 63 Gum tragacanth, 11, 64

Η

Hair creams, 283 Hair milk, 284 Hair tonic, 286

Hair wash, milky, 285 Halowax emulsions, 442 Hand creams, protective, 279 Hay fever ointment, 339 Heat and stability, 71 Heavy ink for spongy leathers, 395 Heel and edge finish, modern transparent, 387 Heel and edge finish, red-brown, 390 Heel and edge stain, dark brown, Hevea latex, 98 Hevea lipin, 16 Hexacosanol, 5 Hexadecanol, 5 High-lustre paper, emulsion for, High melting materials, emulsify-High temperature stable non-aqueous emulsions, 448 Homogenizers, 53 Honey and almond type cream, 269 Hormone cream, 273 Horse power required for mixing, 46 Horse-shoe grease, 326 Humates, 2 Hydrogen ion concentration, (pH), 5, 22, 35, 65, 68, 84, 86, 93, 94 Hydrophilic colloids, 35, 63 Hydrophilic emulsifiers, 63 Hydrophilic emulsions, 63 Hydrophobic emulsifiers, 15 Hydrotropic solvents, 13

Ι

Ice-cream and salad stabilizer, cheese, 299
Icing, butterscotch, 297
Icing, cocoa, 297
Immiscible liquids, emulsification of, 39
Incompatibility, 71
Infant's milk, synthetic, 300
Ink for bottoms, heels and edges, combined, 392
Ink, burnishing, 396

Ink cleaner, 245 Ink, general, 393 Ink, gloss, 396 Ink, one setting edge, 393, 395 Ink, polishing, 393 Ink, safety-paper, 448 Insoluble emulsifying agents, 29 Insoluble powders, dispersing, 450 Instability, causes of, 71 Insufficient dispersion, 71 Interfacial film, electrical charge of, 58 Interfacial tension, 9 Inversion, 17 Invert sugar, 9 Iron, finely divided, 17

J

Japan wax emulsion, 443 Japan wax, ("soluble"), 442

K

Kid cleaner, white, 398 Kier penetrant oil, 426

L

Lacquer-base formulae, (clear), 314 Lacquer emulsifiers, 315 Lacquer emulsion, black pigmented, 318 Lacquer emulsion, gray pigmented, 319 Lacquer emulsions, 313 Lacquer remover for hands, 245 Lanolin cream; boro-glycerin, 272 Lanolin emulsion, 257 Lanolin and lanolin alcohols, 5 Latex, 2 Latex, artificial, 421 Latex, concentrated, 97 Latex emulsions, 97 Laundry wax, self-emulsifying, 443 Lead arabate, 4 Leather cleaner, 245 Leather cleaner, greasy, 245

458 Index

Leather dressings, 321 Leather emulsions, 98 Leather finishes and polishes, 322 Leather polishes, 376 Leathers, heavy inks for spongy, 395 Leather treatment emulsions, 320 Leather, waterproofing for, 435 Lecithin, 2, 10, 16, 21 Lemon juice cream, 273 Lemon oil emulsions, 300 Lemon oil emulsion, transparent, 301 Licorice root, spent, 35 Light brown russet finish, 389 Lime and glycerin cream, 274 Liniments, 335 Linseed extract, 2 Lip make-up, liquid, 274 Liquid esters, 27 Liquid lip make-up, 274 Lotions, skin, 252 Lubricant emulsions, 325 Lubricant, graphite, 327 Lubricant grease, 325 Lubricants, textile, 430 Lubricant, viscous emulsion, 327 Lubricant, water in oil emulsion, 327 Lubricating emulsion, soap-base, 327 Luster emulsion for starching, 426

#### M

Magnesium oleate, 5, 17
Mahogany sulfonates, 8
Mannitol esters, 27
Manton-Gaulin colloid mill, 49
Margarine, 2, 96, 302
Mascara, 274
Massage cream, 280
Mayonnaise, 95
Mayonnaise and salad dressings, 303
Mechanical breaking of emulsions, 78
Medicinal emulsions, 99, 328
Mercuric nitrate ointment, 338

Metal cleaner, 246 Metal drawing compounds, 225 Metal polishes, 399 Metals, protective coating for aluminum and other, 405 Methods of making emulsions, 37 Methyl methacrylate emulsions, 417 Milk, acid skin, 256 Milk, artificial, 1 Milk, bath, 256 Milk, beauty, 256 Milk, hair, 284 Milk, synthetic infant's, 300 Milky hair wash, 285 Mineral oil emulsions, 38, 332 Miscellaneous, 448 Miscellaneous polish emulsions, 403 Miscellanies about mixing, 47 Miscible oils, 92 Miscible oils, formulas of, 216 Miscible oils, naphthenic, 226 Mixed base grease, 326 Mixer speed, 44 Mixing, miscellanies about, 47 Modern transparent heel and edge finish, 387 Montan wax emulsions, 443 Mosquito repelling cream, 280 Moustache pomade, 274 Multi-valent ions, addition of, 19 Mustard, powdered, 19

#### N

Nail cream, 280
Naphthenic acid soaps, 25
Naphthenic miscible oils, 226
"Natural" bottom finish, 387
"Natural leather" bottom stain, 388
"Natural leather" finish, 390
Natural resin emulsions, 417
Neatsfoot oil polish, 412
Nitrocellulose emulsions, 314
Non-aqueous emulsion, 448
Non-foaming oil emulsion, 449
Non-ion active compounds, 106
Non-slippery belt and rope preservative, 430
Nourishing creams, 275

0

Octyl alcohol, 294, 452 Oil, bath, 257 Oil coating for glass fibers, 431 Oil, coconut, 4 Oil, cottonseed, 4 Oil, cutting, 1 Oil, edible, 2 Oil emulsion, almond, 257 Oil emulsions, pine, 286 Oil emulsion, white, 282 Oil, fatty, 8 Oil, fish, 1 Oil, insecticidal, 2 Oil, mineral, 5, 8 Oil, mineral with acacia, 4 Oil, mustard-seed, 4 Oil, Norwegian cod liver, 10 Oil, olive, 4, 5 Oil polish, 412 Oil, poppy-seed, 4 Oils, cutting, 92 Oil, sesame, 4 Oils, miscible, 92 Oil, Turkey red, 10 Oil, vegetable with acacia, 4 Oil-in-water dispersions, 2 Oil-in-water emulsions, 4, 14, 17, 37 Ointment, hay fever, 339 Ointment, mercuric nitrate, 338 Oleates of magnesium, calcium, cobalt, 4 Oleic acid, 17 One setting edge ink, 392, 393, 395 "Opal wax" emulsion, 444 Orange emulsion, (cloudy), 302 Orange oil emulsion, 302 Ouricouri wax emulsion, 445

P

Paint cleaner, soap paste, 246 Painted woodwork cleaner, 246 Paint emulsions, 99, 340 Paints, semi-paste, 345 Pale pink bottom finish, 387 Paper, de-inking printed, 353

Paper, emulsion for high-lustre, Paper processing emulsions, 348 Paper sizings, 348 Paper, waterproofing emulsions for, Paraffin rosin emulsions, 415 Paraffin wax emulsions, 445 Paraffin wax, ("soluble"), 446 Particle size, 5, 64 Particle size of emulsions, 3 Patents on asphalt emulsions, 85 Pectin, 2, 12 Perfume, solidified, 287 Perfumes, water soluble, 287 Peroxide cream, 272 Perspiration deodorizing creams, 267 Petroleum demulsifiers, 449 Petroleum sulfonates, 9 pH, 5, 22, 35, 65, 68, 84, 86, 93, 94 Pharmaceutical emulsions, 99 Phase volume ratios, 61 Phenol-formaldehyde resin emulsion, 419 "Piccolyte" oil emulsion, 346 "Piccolyte" oil emulsion semi-paste, Pine oil disinfectant, 291 Pine oil emulsions, 286 Pine oil soap, 240 Pine tar shampoo, 285 Plasticized vinylite resin AYAA emulsion, 420 Poison ivy lotion, 338 Polar group, relative value of, 58 Polar-nonpolar compounds, 105 Polish, abrasive, 412 Polish emulsions, 100, 354 Polish emulsions, bright-drying rubless floor, 366 Polish emulsions, miscellaneous, 408 Polishes, automobile, 354 Polishes, floor, 364 Polishes, furniture, 371 Polishes, leather, 376 Polishes, metal, 399 Polishes, stove, 406 Polishes, white shoe, 396

Polish, glass, 412 Polishing ink, 393 Polish, neatsfoot oil, 412 Polish, oil, 412 Polish powder, 411 Polyhydric esters of higher fatty acids, 26 Polymerized glycol esters, 28 Polystyrene resin emulsion, 419 Pomade, moustache, 274 Potassium soaps, 4, 25 Powders, dispersing insoluble, 450 Preformed emulsions, 37 Premier colloid mill, 52 Pressure, 43 Printing roll cleaner emulsion, 249 Procedure, improper, 72 Propeller, double, 44 Propeller, single, 44 Propellers, number of, 44 Propylene glycol esters, 28 Protective coating for aluminum and other metals, 405 Protective hand creams, 279 Proteins, colloidal, 61 Psoriasis lotion, 339

Q

Quaternary ammonium salts, 105 Quaternary phosphonium compounds, 105 Quaternary sulfonium compounds, 105 Quick black, 393, 394, 395, 396

R

Radiator anti-foaming agent, 296
Radiator cleaner, auto, 243
Ratio of oil and water, 41
Rayon delustering, 432
Rayon oil, acetate, 431
Rayon, waterproofing, (acetate), 431
Red-brown heel and edge finish, 390

References for emulsifying agents, References for emulsions, 176 Resinates, 9 Resin emulsion, 415 Resin emulsions, natural, 417 Resin oil emulsion, 345 Resin and rubber emulsions, 413 Reversible emulsions, 20 Road emulsions, 86 Rope preservative, 430 Rope preservative, non-slippery belt and, 430 Rosin emulsions, 413 Rosin soap, 17 Rosin turpentine emulsion, 414 Rot-proofing emulsion, 426 Rubber dispersions, 98 Rubber emulsions, 420 Rubber emulsions, chlorinated, 422 Rubber latex, 2, 97 Rubber resin emulsion, 421 Rubber rugs, cleanser for, 250 Rubless floor polish emulsions, bright-drying, 366 Rug cleaners, 248 Russet finish, (buff), wax, 389 Russet finish, light brown, 389 Russet stain, brown, 390

S

Saddle soap, 248 Safety-paper ink, 448 Salad dressings, 306 Salad dressings, mayonnaise and, 303 Salad stabilizer, cheese, ice-cream and, 299 Saponin, 12, 13, 17, 20 Sauce, white or cream, 306 Scouring assistant, textile, 426 Scouring emulsion, textile, 429 Self-emulsifying laundry wax, 443 Semi-paste paints, 345 Shampoo, pine tar, 285 Shampoo, soapless. 285 Shaving creams, 288 Sheep dips, 215

Shoe cleaner, white, 398 Shoe factory use, edge filler for, 324 Shoe ink, 387 Shoemakers' wax burnishing polish, Shoe polishes, white, 396 "Sidol" type polish emulsion, 408 Silicon dioxide, finely divided, 17 Size distribution, determination of, Sizings, paper, 348 Sizings, textile, 423 Skin lotions, 252 Skin milk, acid, 256 Soap, 12 Soap-base lubricating emulsion, 327 Soapless shampoo, 285 Soap paste paint cleaner, 246 Soap, pine oil, 241 Soaps, 24 Soaps, emulsion solvent, 241 Soaps, foaming of, 36 Soaps, formulas of, 239 Soap solutions, 4 Soapy fulling oil, 427 Sodium cetylsulfonate, 17 Sodium dihydroxystearate, 4 Sodium galactonate, 4 Sodium gallate, 4 Sodium glyconate, 4 Sodium kerosene sulfonate, 13 Sodium oleate, 4, 10, 17 Sodium palmitate, 3, 4 Sodium salicylate, 4 Sodium soaps, 4, 25 Sodium stearate, 3, 4 Softeners, textile, 427 Solid esters, 27 Solidified gasoline, 311 Solidified perfume, (oils), 287 "Soluble" oils, 92 Soluble oils, formulas of, 216 Soluble oil, stable to sea water, 220 "Soluble" textile oils, 428 Sorbitol esters, 28 Sound absorbing composition, 312 Spinning oil for light or white fibers, 430

Sports cream, 280 Stability, 5, 56 Stability of emulsions, testing, 73 Stabilizer, cheese, ice-cream and salad, 299 Stabilizer, dairy product and, 299 Stabilizers, 59, 65, 66, 67 Stable non-aqueous emulsions, high temperature, 448 Starch, 1, 9 Starching, luster emulsion for, 426 Sterols, 6 Stokes formula, 59 Stove polishes, 406 Stucco waterproofing, 435 Sucrose, 9 Sugar, 9 Sulfanilamide emulsion, 339 Sulfonated castor oil, 7, 17 Sulfonated herring oil, 8 Sulfonated oils, 6, 7 Sulfonated soybean oil, 8 Sun-burn healing preparations, 283 Sun-burn preventive preparations, Surface-active agents, commercial, Surface tension, 2, 5, 32, 35 Synthetic "cream" for confectionery, 298 Synthetic infant's milk, 300

T

Tannic acid, 2
Tar asphalt emulsion, 42, 238
Tar emulsions, 1
Temperature, 72
Testing stability of emulsions, 73
Tests for determining the emulsion types, 14
Tetrahydronaphthalene and sodium oleate emulsion, 20
Textile dressing, anti-static, 425
Textile emulsions, 423
Textile gloss oil, 427
Textile lubricants, 430
Textile oils, "soluble," 428
Textile scouring assistant, 425

462 Index

Textile scouring emulsion, 429 Textile sizings, 423 Textile softeners, 427 Textile waterproofing emulsions, 433 Tinting dye, fugitive, 426 Tissue cream, avocado, 271 Tragacanth emulsions, 5 Transparent emulsions, 101 Transparent heel and edge finish, modern, 387 Transparent lemon oil emulsion, 301 Triethanolamine oleate, 17 Triethanolamine soaps, 25 Twitchell reagent, 29 Types of emulsions and foams, 14 Types, failure to distinguish, 72

#### IJ

Ultrasonic waves, emulsification by, 40 Univalent electrolytes, 19 Upholstery cleanser, 248 Ursolic acid, 4

#### V

Valerates of ammonium and sodium, 4
Valerates of magnesium and zinc, 4
Vanishing cream, 8
Variations due to formulation and method, 42
Vinylite resin AYAA emulsion, plasticized, 420
Viscosity, 9, 59
Viscosity of colloidal solutions in presence of electrolytes, 60
Viscosity, insufficient, 72
Viscous emulsion lubricant, 327

#### W

Washing compound, 249 Water-dispersible antiseptic oil, 338 Water-in-oil dispersions, 2 Water-in-oil emulsion, 4, 10, 14, 17 Water-in-oil emulsion lubricant, 327 Waterproofing, (acetate) rayon, Waterproofing emulsions, 433 Waterproofing emulsions for paper, Waterproofing for leather, 435 Waterproofing, stucco, 435 Water-soluble perfumes, 287 burnishing polish, shoemakers', 323 Wax emulsions, 102, 436 Wax-resin emulsions, carnauba, 440 Wax russet finish, (buff), 389 Weed killers, 83 Wetting agents, 104 Wetting agents, testing, 105 Whey foams, 35 White bottom cream, 391 White bottom gloss, 391, 397 White or cream sauce, 306 White gloss, 397 White kid cleaner, 398 White oil emulsion, 282 White shoe cleaner, 398 White shoe polishes, 396 Wooden surface cleaner, 249 Wood's metal, 40

 $\mathbf{Y}$ 

Yarn finish, 428

 $\boldsymbol{z}$ 

Zoosterols, 5